# QAPP ADDENDUM (PHASE II) QUALITY ASSURANCE PROJECT PLAN REMEDIAL INVESTIGATION/FEASIBILITY STUDY AMERICAN CHEMICAL SERVICE SITE GRIFFITH, INDIANA

A Supplemental Work Plan (SWP) has been developed for conducting Phase II of the Remedial Investigation/Feasibility Study at the American Chemical Services CERCLA/SARA Site in Griffith, Indiana. The SWP is attached as Appendix A. The original Quality Assurance Project Plan (QAPP) for the project included a scope of work for conducting two Phases of the Remedial Investigation and the the Feasibility Study.

The SWP proposes several modifications and additions to the original Phase II scope of work on the basis of the review of Phase I field observations and analytical results. Sampling location maps and a Phase II activities table are included in the SWP (Appendix A) to summarize the revised Phase II sampling. Table 1 (attached) provides an update to Tables 5 and 6 in the original QAPP, listing the numbers of samples, duplicates, blanks, matrix spikes, and matrix spike duplicates for each sample to be collected in Phase II.

Many of the activities in the modified Phase II scope of work simply increase or re-allocate previously approved sampling procedures; these require no addition to the QAPP. However, some are new sampling procedures which were not in the original Work Plan, and therefore, the QAPP did not include Standard Operating Procedures (SOPs) for performing those analyses. SOPs for the following activities are contained in Appendix B:

- Field screening of VOC concentrations in groundwater.
- Reduction/Oxidation Potential (Redox) testing of field samples while collecting groundwater samples from monitoring wells.
- Dissolved oxygen (DO) testing of field samples while collecting groundwater samples from monitoring wells.
- · Analysis for Total Organic Carbon (TOC) in solid matrix samples.



In addition, laboratory analysis will be used to calculate the total porosity of samples of soils, sediments and aquifer materials. laboratory will follow the procedures in ASTM 0408, (for Soil and Rock), and the Army Corps of Engineers, Laboratory Soils Testing Manual (EM1110-2-1906).

#### **ATTACHMENTS**

Table 1

**APPENDICES** 

Supplemental Work Plan

Standard Operating Procedures
B1 - Groundwater Screening/Soil Gas Sampling Procedure
B2 - Reduction/Oxidation Field Sampling

B3 - Dissolved Oxygen Field Sampling

B4 - Total Organic Carbon Laboratory Analysis



TABLE 1
SUMMARY OF PHASE II SITE CHARACTERIZATION
GAPP ADDENDUM, PHASE II RI
ACS SITE, GRIFFITH, INDIANA

Sample(1) Ma <u>trix</u>	Laboratory	Laboratory Parameters(2,5,6)	Study <sup>(3)</sup> Phase	No. of Investigative Samples	No. of Field <u>Puplicates</u>	No. of Field Blanks	Matrix Spike/ <sup>(4)</sup> Matrix Spike <u>Duplicate</u>	Total No. of <u>Samples</u>
Phase I Monitoring	Compuchem	TCL Volatiles	28	6	1	1	1	9
Wells Round 2	Compuchem	TCL Semi-Volatiles	28	6	1	1	1	9
Groundwater	Compuchem	TCL Volatiles	2A 2B	12 *	2	2	2	18 *
	Compuchem	TCL Semi-Volatiles	2A 2B	12 *	2	2	2	18 *
	Compuchem	TCL PCB/Pesticides	2A 2B	12 *	2	2	2	18 *
	Warzyn	TCL Metals (Dissolved)	2A 28	12 *	2	2	0	16 *
	Warzyn	TCL Metals (Total), TSS	2A 28	5 •	1	1	0	<b>7</b> ★
	Warzyn	Cyanide (Filtered)	2A 2B	12 *	2	2	0	16 *
	Warzyn	Chloride, Alkalinity, Sulfate	2A 2B	12	2	2	0	16 *
	Warzyn	Ammonia, Nitrate-Nitrite TOC, COD	, 2A 28	12	2	2	0	16
	Warzyn	Total Dissolved Solids	2A 2B	12 *	2	2	. 0	16 *
Private	Compuchem	TCL Volatiles	2	10	1	1	1	13
Wells	Compuchem	TCL Semi-Volatiles	2	10	1	!	1	13
(Low Detection	Compuchem	TCL PCB/Pesticides TCL Metals (Total)	2	10 10	1	1	1	13 12
Limits)	Warzyn Warzyn	Cyanide (Unfiltered)	2	10	1	<u> </u>	n	12
r (m( to)	Warzyn	Chloride, Alkalinity, Sulfate,	2	10	i	i	Ŏ	12
	Warzyn	Ammonia, Nitrate-Nitrite	, 2	10	1	1	0	12

TABLE 1 (Continued)

Sample(1) Matrix	Laboratory	Laboratory Parameters(2,5,6)	Study <sup>(3)</sup> Phase	No. of Investigative <u>Samples</u>	No. of Field <u>Duplicates</u>	No. of <u>Field Blanks</u>	Matrix Spike/(4) Matrix Spike Duplicate	Total No. of Samples
Sediment	Compuchem	TCL Volatiles	2	5	1	1	i	8
	Compuchem	TCL Semi-Volatiles	2	5	1	1	1	8
	Compuchem	TCL PCB/Pesticides	2	5	1	1	0	7
	Warzyn	TCL Metals	2	5	1	1	0	7
	Warzyn	Cyanide	2	5	1	1	O	7
Natural Soils	Compuchem	TCL Volatiles#	2	20	2	0	1	23
-Waste Borings	Compuchem	TCL Semi-Volatile #	2	20	2	0	1	23
	Compuchem	TCL PCB/Pesticide:#	2	20	2	0	1	23
	Warzyn	TCL Metals#	2	20	2	0	0	22
	Warzyn	Cyanide#	2	20	2	0	0	22
	Warzyn	Volatile Residue#	2	20	2	0	0	22
Aquifer Matrix	Compuchem	TCL Volatiles	2	5	1	0	1	7
S. Samples	Compuchem	TCL Semi-Volatiles	2	5	1	0	1	7
	Compuchem	TCL PCB/Pesticides	2	5	1	0	1	7
	Warzyn	TCL Metals	2	5	1	0	0	6
	Warzyn	Cyanide	2	5	1	0	0	6
Geotechnical	Warzyn	Atterberg limits	2	12	0	0	0	12
Samples-	Warzyn	Particle Size	2	12	0	0	0	12
Wells and		Total Organic Carbon	2	12	2	0	0	14
Sediment	Warzyn	Cation Exchange Capacity	2.	7	0	0	0	7
	Warzyn	Total Porosity	2	12	0	0	0	12

#### <u>Notes</u>

- 1 Samples will be considered low or medium concentration.
- 2 See Appendix B for TCL analyte lists, also up to 30 tentatively identified compounds.
- The star (\*) indicates that the number of samples and specific parameters will be determined from Phase 1 and 2A results. Preliminary assessment indicates that up to 9 wells will be sampled for the complete TCL, and the remaining number will be sampled for a reduced parameter list. Also note that Phase 2A sample number is given as the expected maximum.
- 4 Sample numbers do not reflect the additional volume of samples required for matrix spikes and matrix spike duplicate analysis.
- Temperature, pH and specific conductance measurements will be taken in the field for aqueous samples. Qualitative screening with the HNu or OVA will be performed on solid samples.
- 6 The # indicates need for each specific analysis will be determined in field.

APPENDIX A

SUPPLEMENTAL WORK PLAN PHASE II RI/FS ACS SITE

( )

Investigation.

## SUPPLEMENTAL WORK PLAN PHASE II REMEDIAL INVESTIGATION AMERICAN CHEMICAL SERVICES SITE GRIFFITH, INDIANA

INTRODUCTION
The original Scope of Work for conducting a Remedial Investigation/
Feasibility Study (RI/FS) at the American Chemical Services CERCLA Site, as
developed for the Work Plan and approved in the consent agreement, specifies
two phases of work for conducting the Remedial Investigation at the ACS
Site. A majority of the Phase I work has been completed. This document
represents a revised scope of work for conducting phase II of the Remedial

In a memo dated October 17, 1989, Robert Swale, U.S. EPA Remedial Project Manager (RPM) proposed an expanded and revised scope of work for conducting Phase II of the RI. The PRP group has spent time and considerable expense during the month which followed Mr. Swale's memo, considering his proposal and responding to it. At the direction of the PRP group, Warzyn Engineering Inc. has drafted this Work Plan Addendum for Phase II RI tasks to respond to the U.S. EPA proposal.

Additional time and expense have been incurred by the PRP group, and the project has been delayed several weeks, while the PRPs have considered the U.S. EPA proposal, and in essence, re-negotiated a scope of work, modified from the one already approved in the signed consent agreement. That original work scope was developed in a joint effort among the U.S. EPA, EPA's technical contractor, Roy F. Weston, and Warzyn (representing the PRP group).

Warzyn has developed this Supplemental Work Plan (SWP) at the request of the PRP group to respond to the U.S. EPA Phase II proposal by re-organizing, modifying, and supplementing the previously-approved Phase II scope of work. If this SWP and associated QAPP addendum can be approved by December 6, 1989, it may be possible to initiate Phase II of the Remedial Investigation during the week of December 11, 1989. The project schedule has been revised for the assumed December II start of Phase II; it is included as Table 1.



#### PROPOSED PHASE II SCOPE OF WORK ACS CERCLA SITE

The following description of activities for the Phase II Remedial Investigation at the ACS CERCLA site is organized in the sequence of the U.S. EPA's October 17, 1989, "Proposal for Phase II of the RI/FS" to facilitate review. The field and sampling activities for each of the activities are summarized in Table 2.

#### A. GROUNDWATER AND SURFACE WATER FLOW DIRECTION

Four monitoring wells will be constructed during Phase II with screens sealed in the lower aquifer. Water levels from these wells will provide data to calculate groundwater flow direction in the lower aquifer. Water levels collected at these lower aquifer wells will be analyzed with the data base which includes water levels at the Phase I monitoring wells, piezometers, and staff gages. The results will be used to document the vertical hydraulic gradient between the upper and lower aquifer across the site. Water levels will also be used to document horizontal gradients in the lower aquifer.

Water levels have been measured at all piezometers, monitoring wells, and staff gages on two dates. Two additional measurements will be made during Phase II of the investigation: one during December 1989/January 1990 when the ground is frozen, and the second during March/April 1990, when the annual hydrograph is expected to be at its peak. Up to four additional water level measurements will be made at a representative group of measuring points (8-12 of the piezometers, monitoring wells, and staff gages), to provide more detailed data regarding interactions between groundwater and surface water, and response to aquifer stresses.

Existing geologic and hydrogeologic information will be evaluated and used to supplement the results of Phase I and II site investigations. Sources include: information in U.S. EPA files, and the <u>Preliminary Hydrogeologic Site Assessment</u> conducted in January 1986 by ATEC Associates for Mr. James Tarpo. The ATEC report contains boring logs, sampling information and water level data from 1986.



A numerical model will be used to synthesize the climatological data, aquifer characteristics data, and water level data into a conceptual flow model. This will be useful in developing an understanding of the groundwater flow system and its interactions with surface water, and in evaluating potential remedial alternative scenarios.

#### B. CONTAMINANT PLUME DELINEATION

Upper Aquifer Investigation. Four to eight Phase II monitoring wells are specified in the approved work plan. Water level measurements in the upper aquifer indicate that there is a groundwater high beneath the ACS Inc. facility, and that groundwater flow may be radially outward. Therefore it is possible that the groundwater plume extends in several directions from the site.

With the potential for a plume to extend in all directions from the site, it is uncertain whether the plume could be adequately delineated if the only further activity is installing eight additional monitoring wells. Therefore, it will be cost effective to use a field screening technique to optimize the locations and limit the number of monitoring wells. Soil gas sampling is a generally accepted field screening technique. However, it appears that the field work will be conducted in the winter when ground will be frozen so there may be potentially high volatile organic concentrations in the ambient air and there may be a high potential of getting meaningless results.

An effective field screening method at the ACS site would be to collect groundwater samples at multiple locations surrounding the site to be analyzed for VOCs or semivolatile compounds. The water levels measured at the piezometer network have provided precise data regarding the depth to the water table, so it would be relatively efficient to penetrate through the water table and collect a groundwater sample for field GC analysis. The result would be an analytical test result for the groundwater at the sampled location.



It is proposed that Tracer Research Corporation (Tracer) be subcontracted to collect water samples from the aquifer in the zones indicated in Figure 1. Tracer uses a sampling van with the capability of driving a sampling probe into the ground to collect a groundwater sample. After the water sample is collected, head space analysis is conducted using the field GC instrumentation in the van. Using this method, it will be possible to map the extent of the VOC plume in the upper aquifer in two or three days. On the basis of the field screening, the well locations will be selected to intersect the outer edge of plume in the upper aquifer, thereby documenting its extent and character. Locations and numbers of wells (up to maximum of eight) necessary to adequately accomplish this goal will be determined from the field screening results.

Aquifer samples (solid matrix samples) will be collected at up to five points within the groundwater contamination plume to provide an indication of contaminant characteristics for remedial alternatives evaluations. The locations will be determined from the field screening results. Parameters for analysis will be VOC and semi-volatile organic compounds.

Lower Aquifer Investigation. The January 1986 Preliminary Hydrogeologic report by ATEC describes a monitoring well constructed in the lower aquifer in 1985. The report indicates that the clay layer is approximately 12 feet thick at the ACS facility, located between elevations 603 and 615 feet mean sea level.

During Phase II, four monitoring wells will be constructed in the lower aquifer to provide hydraulic gradient and water quality information. A double casing drilling technique will be used to avoid potential cross-contamination from the upper aquifer. The wells will be constructed with stainless steel materials, and will have five-foot screens located in the upper zone of the lower aquifer. The first three lower aquifer wells will be constructed at the approximate locations indicated on Figure 2. The fourth well location will be selected to be downgradient of the site on the basis of water levels in the first three.



Groundwater Sampling. The approved work plan specifies that two rounds of sampling will be conducted at each Phase I and Phase II monitoring wells. The target compound list (TCL) of organic parameters and the target analyte list (TAL) for inorganic parameters will be tested for in the first round of sampling at each well. For the second round of sampling at each well, the parameter list may be reduced to test for only the groups of compounds which were indicated in the first round of sampling.

During Phase II, the second round sampling will be conducted at the Phase I wells (MW-1 through MW-6), and both rounds of sampling will be conducted in the Phase II wells. The parameter list for Phase I monitoring wells has been reduced on the basis of Phase I sampling results to include VOCs and semivolatile compounds.

Provided access can be obtained, ten existing water supply wells within one mile of the site will be sampled. Water levels measured in the four lower aquifer monitoring wells will be used to determine the groundwater flow direction in the lower aquifer in the vicinity of the site. Nine downgradient locations and one upgradient location will be selected for sampling and samples will be analyzed for TCL and TAL parameters.

Additional efforts will be made to classify the general characteristics or groupings of the groundwater sampling results which have been classified as "unknown" compounds in Phase I sampling results.

#### C. AQUIFER TESTS AND ENGINEERING EVALUATION

The purpose of conducting aquifer tests in the Remedial Investigation is (1) to provide an adequate characterization of aquifer characteristics to evaluate potential fate and transport of contaminants for the Endangerment Assessment and (2) to provide scoping information for remedial alternatives evaluation in the Feasibility Study.



Aquifer tests were conducted by bail test at each of the Phase I monitoring wells. In addition, grain size analysis was conducted on samples from the aquifer material collected from the screened zone of each of the six monitoring wells. The aquifer tests indicate that the hydraulic conductivity (K) in the upper aquifer ranged between  $1.5 \times 10^{-3}$  cm/sec at MW-2 where the aquifer consisted of fine sand, to  $1.2 \times 10^{-2}$  cm/sec at MW-5, where the aquifer material consisted of sand and gravel.

These results indicate the bail tests, supplemented with grain size analyses, have been adequate to characterize the aquifer properties at the ACS site. There is no indication that conducting a pumping test would provide significantly more precise aquifer data. It is likely that conducting a pumping test would cause delays in project progress because the water pumped during a pumping test would be contaminated. Warzyn's experience indicates that it would be very difficult and time consuming to obtain permits for disposal of the pumped groundwater.

The physical and chemical characteristics of the three major geologic units will be further characterized by additional analyses. Two soil samples will be collected from the upper aquifer (Calumet Aquifer), the confining clay layer, and the lower aquifer (Valparaiso Aquifer). Analyses for each of the six samples will include (as appropriate): grain size, Atterberg Limits, total porosity, and total organic carbon (TOC).

A groundwater flow model will be used to synthesize the slug test data, climatological data, and the water level measurements, and develop a conceptual model of the upper aquifer flow regime.

#### D. FURTHER CHARACTERIZATION OF SITE STRATIGRAPHY

Eight to twelve additional borings will be made to install monitoring wells during the Phase II investigation. Four of the borings will extend through the clay confining layer and be completed as double cased lower aquifer monitoring wells. The results of these boring will document the total thickness of the clay confining layer at different locations beneath the site.



Each of the four lower aquifer wells will be constructed in the vicinity of an upper aquifer monitoring well to create "well nests" at four diverse locations across the site. Water levels measured both above and below the confining layer at each of these locations will provide further data to evaluate the integrity and continuity of the clay layer throughout the site. Additional information regarding physical properties of each geologic unit will be obtained in Activity C.

#### E. DELINEATION OF SURFACE WATER/SEDIMENT CONTAMINATION

The approved Work Plan specified 1 surface water and 1 sediment sample at 11 locations (22 total samples) for the Phase I investigation. During the field activities, there was no standing water at several of the Surface Water/Sediment (SW/SD) sampling locations. Mr. Swale agreed that collecting sediment samples only at these locations would sufficiently characterize the conditions. As a result, the samples which were not collected in Phase I will be re-allocated to Phase II.

Five sediment sampling locations have been identified to further characterize the surficial contamination in the adjacent surface water areas and drainageways surrounding the site and along the railroad between the Griffith Landfill and the marshy area to the north. General locations are shown on Figure 3.

To aid in the determination of the sorptive properties and natural attenuation capabilities of the wetland soils, six near surface soil samples will be collected and submitted for laboratory analysis. Characteristics tests will include grain-size analysis and total organic carbon (TOC) determination.

#### F. WETLANDS DELINEATION

During November 1989, aerial photographs were taken of the ACS site and surroundings. Besides the photography to develop the site base map (1 in = 100 ft, 2-ft contour interval), a black and white photograph and a color infrared photograph were taken. Initial wetland delineation may be conducted through interpretation of these photo maps.



U.S. EPA has reported that there is an interest by the Fish and Wildlife Department, Indiana agencies, and local interest groups, to have detailed wetlands assessment conducted. Apparently, the Fish and Wildlife Department has scheduled site work for the spring of 1990. The PRP group will discuss the requirements of the assessment with the appropriate agencies before the work is scheduled to begin, and develop an approach to avoid duplication of effort.

#### G. TREATABILITY STUDIES

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Phase I findings indicate that there are no wastes or contamination problems which have not been encountered previously at other CERCLA sites. Therefore, there should be existing information from other sites to evaluate the treatability without conducting detailed treatability studies with ACS waste.

To facilitate completion of the feasibility study, it is appropriate to collect some additional data regarding the chemical and physical properties of the contaminated site media. The media which will require remediation are: the soil/waste and the groundwater.

The purposes of Phase II soil/waste sampling are to further delineate the extent of waste (Activity H) and to characterize chemical and physical properties of the waste for compatibility and treatability. Field observations during Phase I indicate that the waste characteristics are highly diverse. The Work Plan specifies that appropriate test parameters may be selected for each sample. The efore, field decisions will be made to perform appropriate analyses for characterizing waste compatibility and treatability. Examples of possible test parameters are total organic carbon, BTU rating, and potential ash generation.

Most of the laboratory analyses which might be useful in assessing groundwater treatability are being conducted in the TCL and TAL sampling required in Round 1 of the sampling. Several field measurements will be conducted during Phase II field work including: pH, temperature, dissolved oxygen, redox potential, depth to groundwater, and saturated thickness.



After the feasibility study is completed, and a final design has been selected, it may be appropriate to conduct bench or scale studies to appropriately scope the final remedy.

#### H. ADDITIONAL WASTE BURIAL DELINEATION AND CLOSING OF DATA GAPS

The approved Work Plan specifies that 20 additional solid matrix samples will be collected during Phase II to further delineate the vertical and horizontal extent of soil/waste contamination at the site. In addition, 10 samples designated for collection in Phase I were not collected. These include the six surface water samples discussed in Activity E (above), and four surface area (SA) samples which were deferred during the Sample Location Staking, conducted on June 15, 1989. (During location staking, representatives of U.S. EPA, U.S. EPA's consultant, and Warzyn agreed to defer surface area samples which were either redundant to other sampling locations, or were located in high traffic areas). Since these samples were Phase I samples, they included the full TCL and TAL parameter list.

As a result, a total of 30 solid matrix samples remain to be collected from the total number of solid matrix samples designated for Phase I and II of the approved work plan. Five of these samples have been allocated to the sampling surface sediment in Activity E above. Another five have been allocated to characterizing the interior upper aquifer contaminant plume (Activity B).

The remaining 20 sampling locations will be assigned to delineate the extent of contamination in known waste areas, and to characterize and delineate waste in newly identified areas. The most flexibility will result by conducting the soil/waste sampling following the procedure used in Phase I.

The Phase I procedure was to delineate the horizontal and vertical extent of buried waste using auger probes; then, to go back to areas which the auger probes indicated were most highly contaminated, or most characteristic of a given area, and collect samples. This procedure will be used in four areas (indicated on Figure 4):



- · A zone between the Kapica area and the Griffith Landfill
- · A zone between the Kapica area and the Off-Site Containment Area
- · The Off-Site Containment Area
- · The area west of the existing treatment lagoon

Auger probes will be used as a field screening method in each area to develop visual and field instrument characterizations of the waste types and the horizontal and vertical extent. Then 20 Phase II solid matrix samples will be used to characterize the waste for compatibility and treatability as appropriate.

#### I. ENVIRONMENTAL AUDIT OF THE AMERICAN CHEMICAL SERVICES FACILITY

The work scope for Phase I of the RI included an environmental audit of the ACS facilities. After a lengthy delay in providing site access to conduct the audit, ACS Inc. management allowed the audit to proceed during the week of November 13, 1989.

#### <u>Tables</u>

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- 1. Phase II Site Investigation Summary
- 2. Round 2 Analytical Parameters for Groundwater Samples

#### Figures

- 1. Field Screening Areas
- 2. Proposed Locations for First Three Lower Aquifer Wells
- 3. Surface Sediment Sampling Areas
- 4. Waste Areas Requiring Further Delineation

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#### TABLE 1

## PHASE II SITE INVESTIGATION SUMMARY AMERICAN CHEMICAL SERVICES SITE REMEDIAL INVESTIGATION

Date	Event/Deliverable
December 6, 1989	Supplemental Work Plan and QAPP Addendum Approval
December 11, 1989	Phase II Mobilization
January 19, 1990	Completion of Phase II Monitoring Wells
February 16, 1990	Completion of Phase II Field Work
March 30, 1990	Validation of Phase II Round 1 samples complete
April 6, 1990	Completion of Phase Round 2 groundwater sampling
June 15, 1990	First Draft Remedial Investigation Report Submitted to U.S. EPA
June 25, 1990	Submittal of Final Remedial Investigation Report to U.S. EPA

Table 2

#### Phase II Site Investigation Summary American Chemical Services CERCLA Site Griffith, Indiana

Activity	Reference <u>Letter</u>	Description	Result	Utilization of Data	Anticipated Number of Investigation Samples
Water Level Measurements	A	Collect water levels at piezometers, monitoring wells, and staff gauges	Additional water level data	Assess hydraulic gradients and surface water gravel water interactions	None
Upper Aquifer Field Screening	8	15 groundwater samples analyzed by head space/field GC	Groundwater samples from upper aquifer	Identify the horizontal extent of groundwater contamination by VOCs	FA 20-30 QC analysis
Monitoring Well Installation Lower Aquifer	B C D	Four monitoring wells screened in lower (Valparaiso) aquifer	Lower aquifer water levels and water samples. Concentration of EPA TCL and TAL parameters	1. Extend site stratigraphy 2. Determine vertical hydraulic gradients 3. Determine horizontal hydraulic gradient	LA: 6 Geotech Samples LA: 4 Analytical Samples IS: Water levels
Upper Aquifer	<b>B</b> ·	Four to eight additional upper aquifer wells installed	Delineate the horizontal and vertical extent of upper aquifer contamination	Document water quality in lower aquifer     Concentration of EPA TCL and TAL parameters	LA: 4-8 Analytical Samples
Aquifer Matrix Sampling	8	Collect 5 aquifer matrix samples from interior of groundwater plume	Concentration of EPA TCL and TAL parameters	Further examine upper aquifer contamination     Data for treatability studies	LA: 5 Analytical samples
Private Well Samplin	ng B	Collect water samples from 1 upgradient and 9 downgradient water supply wells	Concentration of EPA TCL and TAL parameters	Examine for potential contamination of private water supply wells surrounding the site	LA: 10 Analytical samples
Aquifer Tests	C	Permeability testing at 4 upper aquifer and 4 'ower aquifer locations	Estimates of aquifer permeability	<ol> <li>Input to contaminent transport model</li> <li>Dute to calculate fate ad and transport</li> <li>Data to evaluate remedial alternatives</li> </ol>	None
Sediment Sampling	E	Collect 5 mediment samplem from drainageways surrounding site	Concentration of EPA TCL and TAL parameters	Determine if eroding sediments are a potential contamination risk	LA: 5 Analytical

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### Table 2 (continued)

#### Phase II Site Investigation Summary American Chemical Services CERCLA Site Griffith, Indiana

Activity	Reference <u>Letter</u>	Description	<u>Result</u>	Utilization of Data	Anticipated Number of Investigation Samples	
Sediment Sampling	E	Collect 5 near surface soil/ sediment sample.3		Determine sorptive properties and natural attenuation capabilities of wetland	LA: 5 Geotechnical samples	•
Aerial Photograph	F	Infrared color aerial photo taken of site	Infrared color photo	Wetland delineation	None	
Field Parameter Analysis	G	,	monitoring well water	Aid in assessing treatability of groundwater	IS: 18	•
Waste Sampling	н	Collect 20 additional waste/soil samples	Physical and chemical extent of buried waste	Data for compatibility and treatability evaluator	LA: 20	,

#### Notes:

IS = Insitu Analysis

LA = Laboratory Analysis

FA = Field Analysis

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TANK FARM

PALFOAD FENCE

BUILDING DIRT ROAD

MWI+

EXISTING PHASE I MONITORING WELL LOCATION & NUMBER

AREAS TO BE SCREENED FOR EXTENT OF GROUNDWATER PLUME

150 SCALE (FEET)

ABANDONED RALROAD

NORTH

FIGURE 1

	į	FIELD SCREENING AREAS	WARZYN	V28	ELR	"TWP
JE SI		SUPPLEMENTAL WORK PLAN	distant packet tank and Materia - Minister Minister - Minister	- CYAVA		- whylm
43 6		PHASE II REMEDIAL INVESTIGATION AMERICAN CHEMICAL SERVICES SITE				***************************************
1 0		GRIFFITH, INDIANA				
	•			4 MH - Spring (spring to - 22		

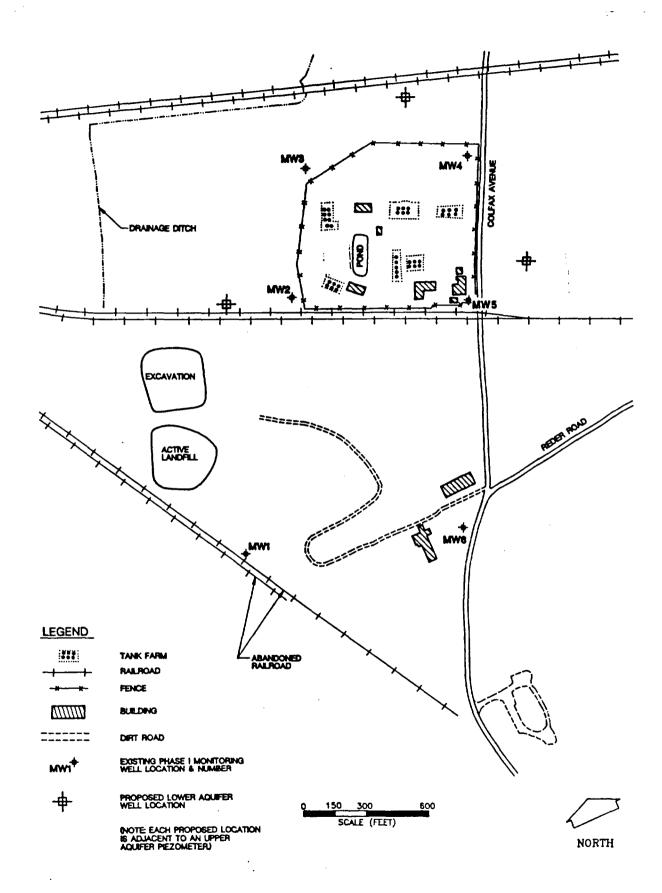


FIGURE 2

		PROPOSED LOCATIONS FOR FIRST THREE LOWER AQUIFER WELLS	NARZYN	 - ELR	and TWP
45 8		SUPPLEMENTAL WORK PLAN	MANUAL PROPERTY OF	 	11/22/11
	1	PHASE II REMEDIAL INVESTIGATION AMERICAN CHEMICAL SERVICES SITE			
13 8	·	GRIFFITH, INDIANA			
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#### FIGURE 3

SUPPLEMENTAL WORK PLAN PHASE II REMEDIAL INVESTIGATION AMERICAN CHEMICAL SERVICES SITE GRIFFITH, INDIANA			•					
SUPPLEMENTAL WORK PLAN PHASE II REMEDIAL INVESTIGATION AMERICAN CHEMICAL SERVICES SITE			Ī	SURFACE SEDIMENT SAMPLING AREAS	WARZYN	13v	ELR	come TWP
1 Self-Step (groups or - or form bond	4	0251.0	OF	PHASE II REMEDIAL INVESTIGATION	Printer - State of the later of	74		וארואי

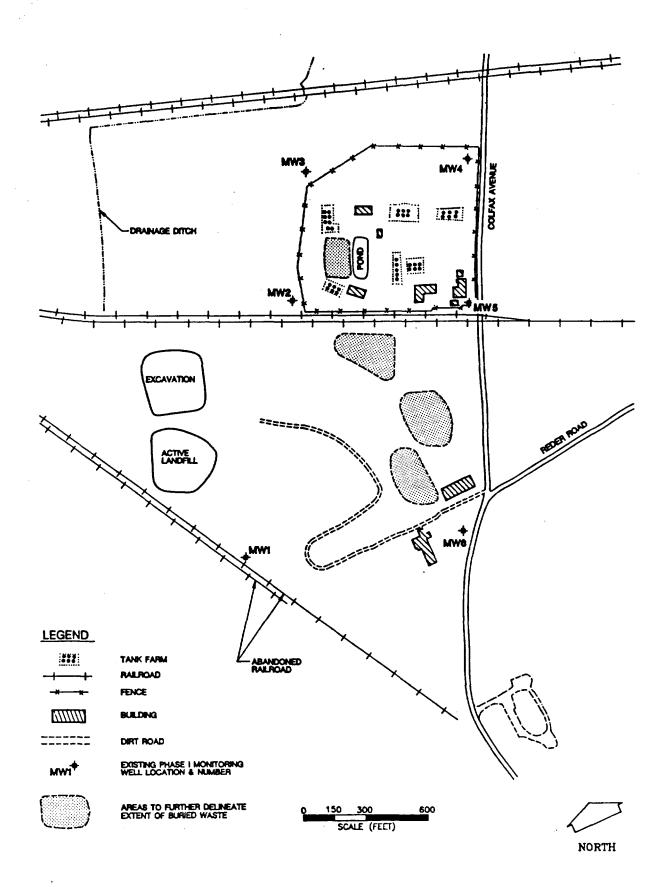
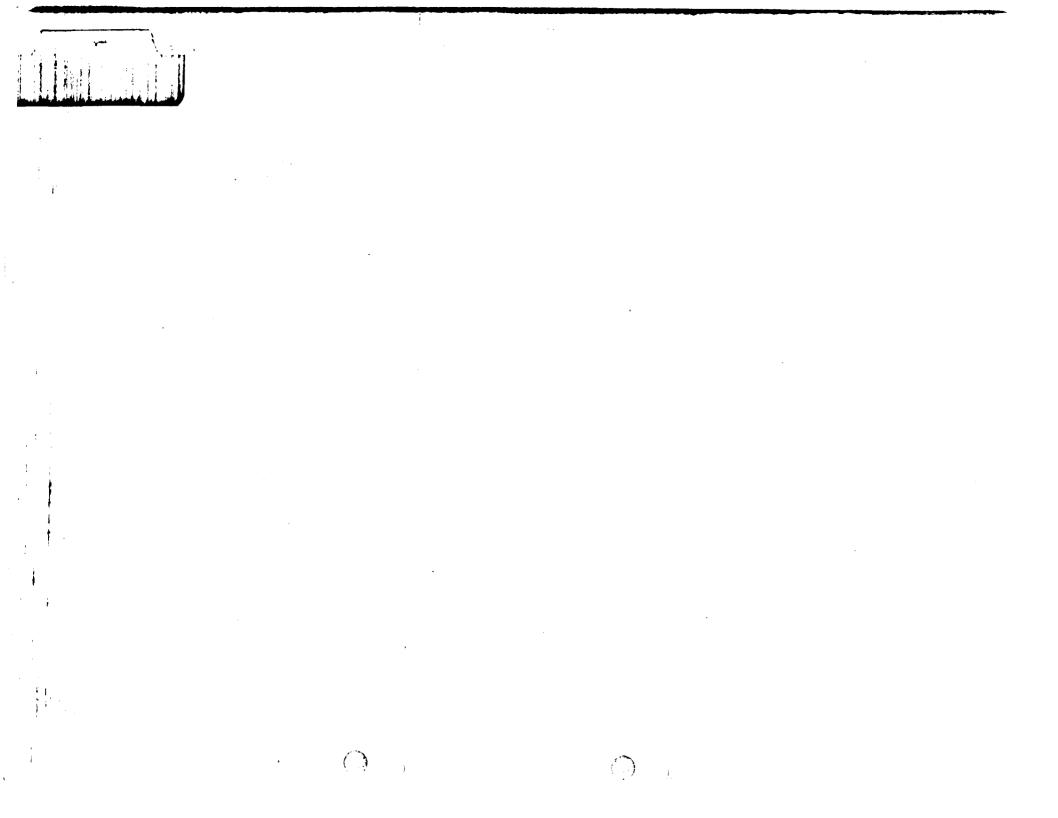


FIGURE 4

1	I	WASTE AREAS REQUIRING FURTHER DELINEATION	WARZYN	مع. م	ELF	Twp
80251.08 WARTIN	워기	SUPPLEMENTAL WORK PLAN PHASE II REMEDIAL INVESTIGATION AMERICAN CHEMICAL BERVICES SITE GRIFFITH, INDIANA	SHAIM HEAD LINE DE SHAIP - HEAD Mark gas - Ousp Shris gas - Ousp Shris	57h	Ages Residue	11/20/21

APPENDIX B
STANDARD OPERATING PROCEDURES



APPENDIX B1
GROUNDWATER SCREENING/SOIL GAS SAMPLING PROCEDURE SOP

#### APPENDIX B1

GROUNDWATER SCREENING/SOIL GAS SAMPLING PROCEDURES
TRACER RESEARCH CORPORATION



#### A. METHOD

Probes driven are into the ground by the pusher/puller mechanism. If there is concrete or pavement over a sample location, TRC personnel use a Kango hammer drill to drill a 1-1/2" diameter hole through the surface material. useful for going through up to 2" of concrete or 10" of asphalt. After 3-5 probe volumes have been drawn through the probe using a vacuum pump, a gas sample is taken by a glass syringe which is inserted through a section of silicone tubing (leading to the pump) and into the stainless steel tubing in the adaptor (Figure Gas samples only contact steel surfaces and are never in contact with potentially sorbing materials (i.e. tubing, hose, pump disphragm). A vacuum gauge monitors the negative pressure in the evacuation line to assure that there is no impedance to gas flow caused by clayey or water-saturated soils.

Three 10 ml air samples are collected from each sampling probe after 1 to 4 minutes of pumping. These 10 ml samples are subsampled according to analytical requirements and replicates are injected into the gas chromatograph for documentation of reproducibility. More than two injections may be necessary where there are multiple contaminants which require different sample sizes for chromatographic analysis. TRC has determined that reproducibility of soil gas samples from the same probe is typically within 20% and always within a factor of two. sampling error is well within the limits required to accurately map concentration contours in the vadose zone which normally range 3 to 6 orders of magnitude over a subsurface plume. Correlation coefficients between contaminant concentrations in soil gas and in groundwater are determined by sampling probes near existing monitor wells and are interpreted on an order-ofmagnitude basis.

FIGURE 1. SAMPLING APPARATUS

10. CLOSE-UP OF SYRINGE SOIL GAS SAMPLING THROUGH EVACUATION LINE 18. DIAGRAM OF SOIL GAS SAMPLING PROBE WITH ADAPTOR FOR SAMPLING AND EVACUATION OF THE PROBE AFTER IT 15 DRIVEN INTO THE GROUND

Once sampling has been completed, the probe is withdrawn and backfilled with native soil or granulated bentonite. Asphalt or concrete patch is used to cap holes that have been driven through paved or concrete areas.

In the event the van cannot be driven to a sample location, sampling probes can be hand-pounded into place and sampled using remote or battery operated equipment.

#### B. CHECKS FOR CONTAMINATION

Prior to sampling each day, system blanks are run to check the sampling apparatus (probe, adaptor, 10cc syringe) for contamination by drawing ambient air from above ground through the system and comparing the analysis to a concurrently sampled air analysis. System blanks are repeated after approximately every 10 soil gas sampling locations.

#### C. SAMPLING EQUIPMENT DECONTAMINATION

- Steel probes are used only once during the day and then washed with high pressure soap and hot water spray or steam-cleaned to eliminate the possibility of cross-contamination. 42 probes are carried on each van to avoid the need to reuse any during the day.
- Probe adaptors (steel reducer and tubing) are used once during the course of the day and cleaned at the end of each working day by baking in the GC oven. The tubing is replaced periodically as needed during the job to insure cleanliness and good fit.
- silicone tubing (connecting the adaptor to the vacuum pump) is replaced as needed to insure proper sealing around the syringe needle. This tubing does not directly contact soil gas samples.



#### ANALYTICAL CHEMISTRY PROCEDURES

#### A. METHOD

Halocarbon and hydrocarbon compounds detected in soil gas are identified by chromatographic retention time. Verification of compound identity is obtained by chromatographic analysis with columns of differing polarity and selectivity.

Quantification of compounds is achieved by comparison of the detector response of the sample with the response measured for calibration standards (external standardization). Instrument calibration checks are run periodically throughout the day as are system blanks to check for contamination in the soil gas sampling equipment. Air samples are also routinely analyzed to check for background levels in the atmosphere.

Proprietary modifications to the gas chromatograph allow direct aqueous injections of water for analysis. Results of both soil gas and water injection analysis are available to the site engineer within 30 minutes of sample collection.

#### B. CHECKS FOR CONTAMINATION

- 2 cc subsampling syringes are checked for contamination prior to sampling each day by injecting nitrogen carrier gas into the gas chromatograph
- . Microliter size subsampling syringes are reused only after a nitrogen carrier gas blank is run to insure it is not contaminated by the previous sample

#### C. ANALYTICAL EQUIPMENT CALIBRATION

At the beginning of each day, standards are analyzed to calibrate the analytical equipment and determine daily response factors. Chemical standards are prepared in water from commercially available pure standards stored in methanol. Prior

to running standards, water for standards is analyzed for purity. At least three standard injections are analyzed until resultant responses fall within 25% of each other. Response factors are then calculated based on these standard responses. Standards are repeated after every 5 samples to verify response.

#### D. ANALYTICAL PROCEDURES

Samples are collected in 10 ml glass syringes and subsampled for analysis in volumes ranging from 1 ul to 2 ml. Injection volume is varied to insure that resultant masses of analyte fall within the linear response range of daily standards. All subsampling syringes and needles are used only once before decontamination.

#### E. DETECTION LIMITS

Detection limits are a function of the injection volume as well as the detector sensitivity for individual compounds. Thus, the detection limit varies with the sample size. Generally, the larger the injection size the greater the sensitivity. peaks for compounds of interest must be kept within linear range of the detector. If any compound has a high concentration, it is nccessary to use small injections, and in some cases to dilute the sample to keep it within linear range. This may cause decreased detection limits for other compounds in the analyses. The detection limits range down to 0.01 ug/L in soil gas for compounds such as benzene and 0.00005 ug/L in soil gas for compounds such as carbon tetrachloride depending on conditions of the measurement, in particular, the sample size. If any component being analyzed is not detected, the detection limit for that compound in that analysis is given as a "less than value (e.g. <0.1 ug/l). This number is calculated from the



#### DOCUMENTATION

-

A numbering system for soil gas is established prior to sampling and remains consistent throughout each phase of an investigation. Because chemical analyses are performed on-site, conventional chain of custody protocols are unnecessary. There are no soil gas samples to loose or preserve. Water samples are immediately labeled with the date, time, depth and location number of each probe. The probe location number is entered on each chromatogram and verified by TRC's field personnel. operator is responsible for checking and interpreting each day's The field assistant is responsible for plotting chromatograms. probe locations on the .map and entering the date, time and location number of sampling points into the log book. of contaminant concentrations for each location are compiled on TRC data sheets by the GC operator and checked by the field assistant. The standards and response factors used for calculations will be present on the same sheet with the sample data calculated from them. Each time during the investigation that the instrument is recalibrated, a new data Thus, it will always be clear which sheet will be started. standards are used for each calculation.

#### A. FIELD DATA SHEETS

An example field data sheet is attached to show how all pertinent information is recorded. The data sheets were designed to contain all the information needed to access the original chromatograms and to check every aspect of the calculations. The documentation as well as other QA procedures have been developed to satisfy the needs of EPA Superfund and other investigations where it is anticipated that the data may be exposed to legal scrutiny.

(



#### B. CHROMATOGRAMS

The GC operator will document each set of chromatograms with the following information:

- Gas flows for  $H_2$ ,  $N_2$ , and air Tank pressures for  $H_2$ ,  $N_2$ , and air 2.
- Temperatures
  - a. injector
  - column b.
  - detector
- Integrator Parameters
  - injector a.
  - peak markers ъ.
  - baseline offset c.
- Column
  - 8. type
  - b. length and diameter
  - c. packing material
  - temperature
- Operator
- Date 7.

If any system parameters change, the GC operator will document on the chromatograms that the changes occurred, and will list the actual changes on the chromatograms.

#### LOG BOOK

The field operator assistant will maintain a daily log book as well as individual field logs for each sample location ranording the following information for each sample location:

- 1. Time (military notation) and weather
- Ambient air and soil temperature 2.
- Sample number (determined by client)
- Location (keyed to mapped location supplied by client and an approximate description, including street name)
- 5. Sampling depth
- Evacuation time between samples 6.
- Flowrate (milliliters per minute)
- Probe and adaptor numbers and volume of the sample probe 8.
- Number of sampling points used 9.
- Observations (including, but not limited to: ground 10. conditions, concrete, asphalt, soil appearance, surface water, odors and vegetation)
- Backfill procedure and materials 11.

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                                                              Column TEMP. Iso 100 °c Program ...
                FUNCTION
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                   50. 21
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FIGURE 4. CHROMATOGRAM DOCUMENTATION

2, 15

### SOIL GAS INVESTIGATION BACKGROUND INFORMATION

STIE NAME: 21	VIDSON CHEMICAL
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INTES OF INVEST	IGATION: 2/16-2/16/88
ditent name & a	ADDRESS: BRANDENBURG CONSULTANTS
	602 MALISTN ROAD
	WHERENMHAL SO 19657
LITIO PRESENTAT	TIVE(S) FOR CLIENT: TOSAN PANDENET
	REPORT AND QUESTIONS
ENOULD BE DIE	PHONES (183 ) 972-1003 EXT. 5
	PHONE: (7/3) 972-1005 Ext. 5
CHEWIST	S. KULLER GEOLOGIST M. FAVERON
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FIGURE 2A. FIELD LOGBOOK - BACKGROUND INFORMATION

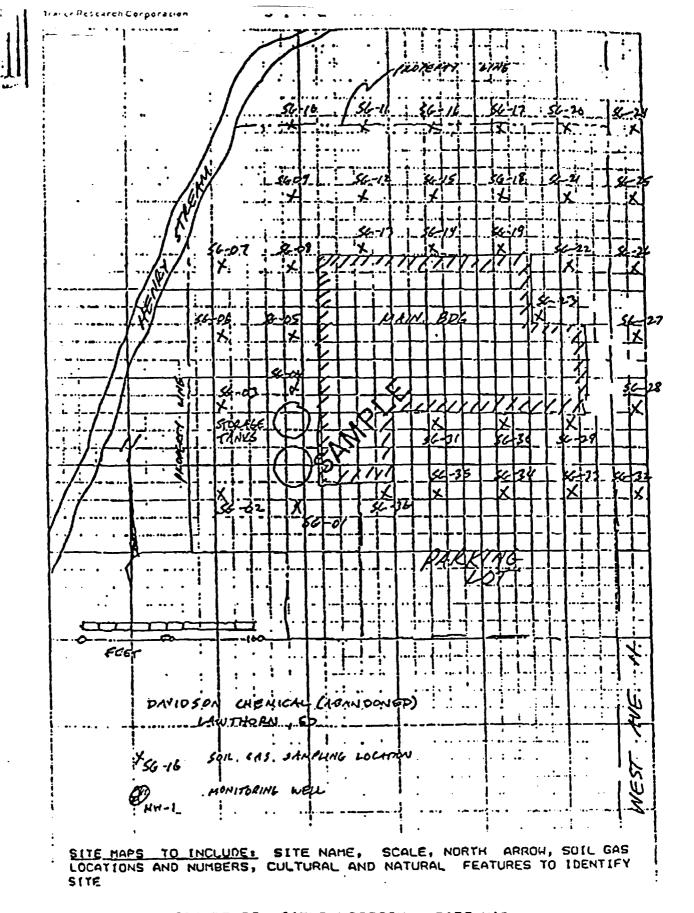


FIGURE 28. FIELD LOGBOOK - SITE MAP

DATE : 2-16-85 LOCATION: PAVIPSON CHENICAL CLIENT I BRANDENBURG

Chamist : S Kupien Weather 1 12"; CLEAR & COLD Geologist : M. FAVERON

FIELD HOURS\_

Mime on site : 730 Time off site : 1750 Lunch hours " Downtime hours' : Ø Standby hours : 0

Hours on site : 10 ( - - )

\_D E C O N T A M I N A T I O N,

Probe Decontamination

Time start : 1745

Time end

Total hours:

Varified by geologist.

Syringe Decontamination

Time start 1 2000 Time end : 2050

Total hours :

Cverified by chemist

\_\_ D A I L Y SUMMARY\_\_\_\_

Calibration

Time start: 730

Time and : 830

Total hours:

Sampling

Max vacuum : 23 in Hg Probes used :/8

Points used : 20 Soil gas samples

collected :/8 Water samples

collected : P

Field data and gas standards checked by Data checking hours: //a

Analysis

:/

: 3

Total system

blanks

Total air samples

' - Downtime includes time spent repairing sampling & analytic equipment; note times and explanation on following field data pages

" - Standby includes time available for sampling but waiting for client; note times and explanation on following field data pages

FIGURE 2C. FIELD LOGBOOK - DAILY SUMMARY

1		٠	<b>~</b> ,		8 A	M	LIN	g	D	ATA
Ę	SAMPLE	DEPTH	PROBE#	ADAPT#	PROBE PUSH/ POUND	VAC(In Hg)	EVAC TIME (9)	SAMPLE VOL(ec)	POINTS	NOTES/ADD'L DATA . REQUESTED BY CLIENT
	SYSTEM BLANK	M	16	20/	M1	,	<i>3</i> 0,	Ece	KA	
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	56-02	6'	30/	16	MA TO L	5	303	珍の	,	ASTANT IS ECHAPED TO THE CARTERY; SIST AND PUBLICANTE ON APPLICANCE; NO NECO TO DEAL; PASS ONTERED EATHY
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				-	-		-	<u> </u>	-	
	•			;		:	:		:	

FIGURE 20. FIELD LOGBOOK - SAMPLING DATA

- 12. Actual sample location marked on the site map (1 inch = 300 feet) provided by client Barometric pressure
- 13.
- 14. Relative degree at condensation in duplicate sample container

#### D. DAILY REPORT

Two hours before the end of each day's work, the GC operator supplies the on-site client representative with a condensed copy of the day's analyses plus condensed data for the previous day's This data, in addition to a map of the sample locations, will constitute TRC's daily report to the client.

#### QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES

complete Research Corporation has Assurance/Quality Control Program for its soil gas contaminant investigation services. Included as part of this is a full Field Operation Manual with very detail QA/QC procedures. outlined below summarize TRC's overall QA/QC program.

If needed a client can be provided with documentation detailing the entire program.

# Reusable Sampling Equipment

- Steel probes are used only once during the day and then washed with high pressure soap and hot water spray or steam-cleaned to eliminate the possibility of crosscontamination. 42 probes are carried on each van to avoid the need to reuse any during the day.
- Probe adaptors (steel reducer and tubing) are used once during the course of the day and cleaned at the end of each working day by baking in the GC oven. The tubing is replaced periodically as needed during the job to insure cleanliness and good fit.



- silicone tubing (connecting the adaptor to the vacuum pump) is replaced as needed to insure proper sealing around the syringe needle. This tubing does not directly contact soil gas samples.
- Glass syringes are usually used for only one sample per day and are washed and baked out at night. If they must be used twice, they are purged with carrier gas (nitrogen) and baked out between probe samplings.
- Septa through which soil gas samples are injected into the chromatograph are replaced on a daily basis to prevent possible gas leaks from the chromatographic column.
- Analytical instruments are calibrated each day by the use of chemical standards prepared in water by serial dilution from commercially available pure chemicals. Calibration checks are also run after approximately every five soil gas sampling locations.
- 2 cc sampling syringes are checked for contamination prior to sampling each day by injecting nitrogen carrier gas into the gas chromatograph
- Prior to sampling each day, system blanks are run to check the sampling apparatus (probe, adaptor, 10 cc syringe) for contamination by drawing ambient air from above ground through the system and comparing the analysis to a concurrently sampled air analysis. System blanks are repeated after approximately every 10 soil gas sampling locations.
- All sampling and 2 cc subsampling syringes are decontaminated each day and no such equipment is reused before being decontaminated. Microliter size subsampling syringes are reused only after a nitrogen carrier gas blank is run to insure it is not contaminated by the previous sample.

Soil gas pumping is monitored by a vacuum gauge to insure that an adequate gas flow from the vadose zone is maintained. A negative pressure (vacuum) of 2 in. Hg less than the maximum capability of the pump (evacuation rate >0.02 cfm) usually indicates that a reliable gas sample cannot be obtained because the soil has a very low air permeability.

All contaminated sampling equipment (probes, adaptors, syringes) is stored separately from clean equipment to prevent cross-contamination and accidental re-use.

.

APPENDIX B2
REDUCTION/OXIDATION FIELD SAMPLING SOP

ORION
Laboratory Products Group
Orion Research Incorporated

# MODEL 230 INSTRUCTION MANUAL

# CONTENTS

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# **GENERAL INFORMATION**

#### introduction

The ORION Model SA 230 is a portable battery-operated digital pH meter for field, plant or laboratory use. It is designed for versatile, easy operation, and can be used in or out of the accompanying carrying case. The instrument is lightweight and designed to fit comfortably into the hand. All controls are on the meter face which affords one hand calibration.

The instrument measuring range is -1999 to + 1999 mV, and pH is displayed to two decimal places. The meter has a large easy to read LCD display.

#### Instrument Description See Figure 1.

- On/Off switch Slide switch controls power to meter.
- 2 LCD display Model SA 230 automatically displays data in large numerals with negative polarity sign and decimal point. pH values are displayed from 0 to 14 with 0.01 pH unit resolution. Millivolt range is –1999 to + 1999. Dissolved oxygen is measured from 0 to 14 ppm when meter is used with 97-08 Oxygen Probe. Temperature is displayed in °C with 0.1°C resolution.
- 3 Mode Control Provides operator with choice of sample measurement in either pH or mV mode, and temperature in °C.
- 4 Calibration Control Used to standardize the meter/electrode system in buffers of known pH.
- 5 Temperature/slope Control Compensates for variation in electrode slope or solution temperature.
- 6 Electrode Connections Accepts BNC connector from combination electrodes and pin tip jack available for use with separate half-cell reference electrodes. Accepts standard ATC Banana plug connector.

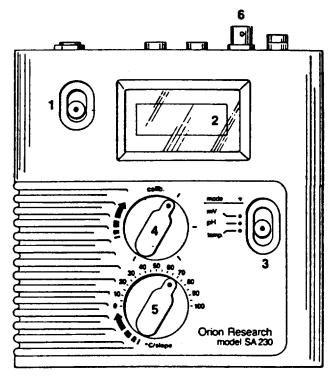


Figure 1 Face of the SA 230

# **INSTRUMENT SET-UP**

#### **Support Rod**

- Attach support rod base to side of meter carrying case and tighten clamp screw.
- Insert support rod into base. Tighten by turning rod clockwise.
- 3. Attach electrode holder to top of support rod.

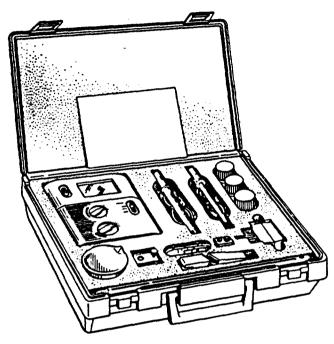


Figure 2
Model SA 230 and accessories

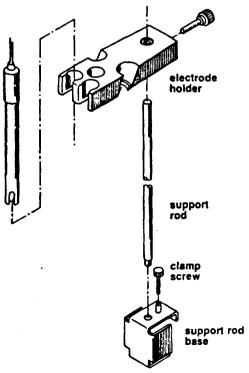


Figure 3
Support rod and clamp

#### **Power Source**

ORION Model SA 230 operates on one non-rechargeable 9-volt alkaline battery. Optional AC line adapters are available for both 110 and 220 volt mains. Refer to ACCESSORIES, page 7.

#### Battery Installation See Figure 4.

- Remove access panel on the back of meter. No tools are required, simply slide cover towards bottom of meter.
- 2. Attach battery connector clip of meter to battery terminals, install battery and replace access panel.

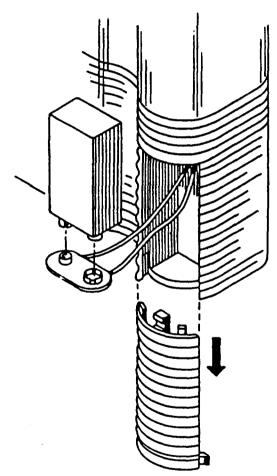


Figure 4 Rear access panel removed

#### **Electrode Connections**

- 1. Attach electrodes with BNC connectors directly to the top of the meter. Refer to **Figure 5**.
- Attach electrodes with U.S. Standard connectors, using ORION Cat. No. 090033 U.S. Standard to BNC adapter. See Figure 6.

If using a combination electrode, electrode connection 4 is not used.

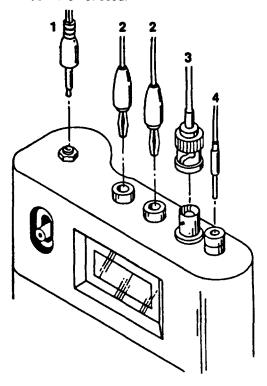


Figure 5
Electrode connections

#### Legend

- 1 AC line adapter
- 2 ATC probe connectors
- 3 BNC connector
- 4 reference pin-tip connector

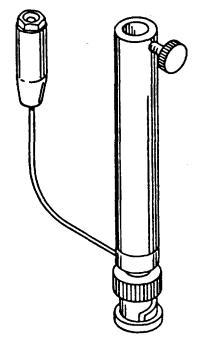


Figure 6
U.S. Standard connector to BNC adapter

#### **Meter Check-Out Procedure**

- 1. Slide power switch to ON position.
- 2. If using optional AC line adapter, connect it to meter and appropriate power source. Proceed to step 4.
- 3. Slide mode switch to pH. If LO BATT indicator on LCD remains on, battery must be replaced.
- Attach BNC shorting plug (ORION Cat. No. 090045) to BNC connector on top of meter. Slide mode switch to pH. Adjust calib knob to read a steady 7.00. If this cannot be done, refer to TROUBLE-SHOOTING, page 6.
- Remove the shorting plug. After successful completion of steps 1-4, the meter is ready for use with an electrode.

# **MEASUREMENT PROCEDURES**

#### pH Measurements

#### Single Buffer Standardization (Without ATC)

- Sample and buffer temperature must be equal. Set temp/slope control to buffer temperature (°C).
- Place electrode in a buffer solution with pH value within 1.5 units of the expected sample value and stir moderately.
- Slide mode switch to pH. Allow reading to stabilize, then adjust calib control so that correct buffer value at that temperature is displayed.
- Remove electrodes from the buffer solution, rinse, and place electrodes into sample. Stir moderately. Allow reading to stabilize.
- 5. Record pH value displayed.

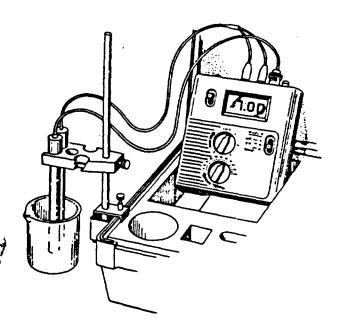


Figure 7 SA 230 meter set up for sample measurement

#### Single Buffer Standardization (With ATC)

The ATC probe replaces the manual temp/slope control on front panel with automatic compensation based on sensed sample temperature.

- Plug ATC probe into input jacks. Slide mode switch to temperature. Verify that ambient temperature is displayed.
- Place electrodes in a buffer solution with pH value within 1.5 units of the expected sample value and stir moderately.
- Slide mode switch to pH. Allow reading to stabilize, then adjust calib control so that pH value of buffer is displayed.

- Remove electrodes from the buffer solution, rinse, and place electrodes into sample. Stir moderately. Allow reading to stabilize.
- 5. Record pH value displayed.

#### Two Buffer Standardization (Without ATC)

NOTE: For maximum accuracy, perform a two buffer calibration once at the beginning of each day. This procedure provides the correct setting for temp/slope control. Subsequent measurements are made after a single buffer calibration.

- Sample and buffer temperature multi be equal. Set temp/slope control to buffer temperature (°C).
- 2. Place electrode in pH 7 buffer and stir moderately.
- Slide mode switch to pH. Allow reading to stabilize, then adjust calib control so that correct buffer value at that temperature is displayed.
- Remove electrode from the buffer solution, rinse, and place electrode in second buffer. Stir moderately. Allow reading to stabilize.
- Adjust temp/slope control until correct value of second buffer is displayed. Remove electrode from solution, rinse and place electrode in sample. Stir moderately. Allow reading to stabilize.
- 6. Record pH value displayed.

NOTE: If sample temperature differs significantly from the buffer temperatures used to calibrate, an adjustment can be made. Raise or lower the temp/slope control from its current setting, described in step 6, by the difference between the actual buffer temperature and the sample temperature. For maximum accuracy use ATC probe.

#### Two Buffer Standardization (With ATC)

- Plug ATC probe into input jacks and adjust temp/slope control knob to 25°C. Slide mode switch to temperature. Verily that ambient temperature is displayed.
- 2. Place electrodes in pH7 buffer and stir moderately.
- Slide mode switch to pH. Allow reading to stabilize, then adjust calib control for correct display value.
- Remove electrodes from the buffer solution, rinse, and place electrodes in second buffer. Stir moderately. Allow reading to stabilize.
- Adjust temp/slope control until correct value of second buffer is displayed. Remove electrodes from solution, rinse and place electrodes in sample. Stir moderately. Allow reading to stabilize.
- 6. Record pH value displayed.

#### **Potentiometric Measurements**

Potentiometric titrations are performed in mV mode using either ion selective or redox electrodes with BNC connectors. Detailed instructions for any ORION electrode are given in the electrode instruction manual. Titration instructions are included in ORION Redox Electrode (Model 96-78) Instruction Manual, or in standard analytical chemistry texts. U.S. Standard to BNC adapters are available from ORION (Cat.No. 090033).

#### **Dissolved Oxygen Measurement**

Dissolved oxygen measurements are displayed in ppm when ORION Model 97-08-99 Dissolved Oxygen Electrode is used with Model SA 230. Follow the instructions in the electrode manual. Be sure to set meter mode switch to pH.

# TROUBLESHOOTING GUIDE

The following section covers troubleshooting that can be performed without special tools or skills. The ORION Technical Service Personnel can be consulted for troubleshooting advice by calling 1-800-225-1480 or 617-864-5400. Outside North America contact your local authorized ORION Representative.

Malfunction	Possible Cause	Remedy
No Display	No power to meter	Check that switch is in ON position.
•	•	Replace battery.
		Check that adapter is receiving power and is plugged in securely.
More than one decimal displayed	mode switch is between positions	Adjust switch to proper position.
Erratic readings or drift. Readings out of range.	Liectrode failure	Follow meter checkout procedure. If meter okay, check electrode.

### INSTRUMENT WARRANTY

ORION RESEARCH INCORPORATED warrants this instrument will operate for one year from the date of purchase when used under normal laboratory conditions, and in accordance with the operating limitations and maintenance procedures given in the instruction manual. In the event of failure within the warrant period, ORION, or its Authorized Dealer, will, at ORION'S option, repair or replace the non-conforming instrument at no charge to the customer.

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# **OPTIONAL ACCESSORIES**

Description

Cat. No.

020121 220V AC line adapter 020041 Shoulder strap and meter holder for hands free operation 020042 Carrying case with foam insert, without meter or accessories 020043 Support rod and guide 020044 Accessory pack includes two 60 ml bottles and one 150 ml beaker	<b></b>	
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### **SPECIFICATIONS**

Model SA 230, ORION Cat. No. 023000 portable pH/mV/temperature meter for hand held and bench top use. Digital, LCD meter comes in carrying case with combination pH electrode, ATC and accessories, ready for immediate use. 110 and 220 volt adapter(s) available for AC line use.

#### Modes

pH, mV, temp, O<sub>2</sub> (with O<sub>2</sub> probe) --

pH range

pH 0 to 14

pH resolution

0.01

mV range

-1999 to +1999 mV showing negative polarity sign

mV resolution

1 mV

Temperature compensation

Automatic and manual

Sample temperature range

-5 to 105°C

Resolution of sample temperature

0.1°C

Input impedance

>100,000 megohms

Instrument drift

<50 microvolts/°C

Input bias current

 $< \pm 1$  pico amp at 25°C and  $< \pm 4$  pico amps over full operating range

**Environmental requirements** 

5 to 45°C and 5 to 80% relative humidity, non-condensing

Isopotential point

pH 7 (fixed)

Power requirement

One 9 volt battery. Optional 110 or 220 volt line adapter(s) available for AC line use.

Inputs

BNC combination and separate pin tip reference jack

Meter dimensions

14 cm X 14 cm X 4 cm

Meter weight

0.5 kg

Meter case

Splash-proof, chemical resistant

Carrying case dimensions

38.1 cm x 27.9 cm x 11.4 cm

Carrying case weight

1.8 kg

# NOTICE OF COMPLIANCE

This meter may generate radio frequency energy and if not installed and used properly, that is, in strict accordance with the manufacturer's instructions, may cause interference to radio and television reception. It has been type-tested and found to comply with the limits for a Class B computing device in accordance with specifications in Subpart J of Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference in a residential installation. However, there is no guarantee that interference will not occur in a particular installation. If the meter does cause interference to radio or television reception, which can be determined by turning the unit off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- Reorient the receiving antenna.
- Relocate the meter with respect to the receiver
- Move the meter away from the receiver.
- Plug the meter into a different outlet so that the meter and receiver are on different branch circuits.

If necessary, the user should consult the manufacturer or an experienced radio/television technician for additional suggestions. The user may find the following booklet prepared by the Federal Communications Commission helpful:

"How to Identify and Resolve Radio-TV Interference Problems."

This booklet is available from the U.S. Government-Printing Office, Washington, D.C. 20402, Stock No. 004-000-00345-4.

# **ORION**

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Form No. 2301M/4811(A)

Printed in U.S.A.



#### E-11

#### Platinum Combination Electrode

#### Catalog No. 13-639-82

The Fisher platinum combination electrode combines a silver/silver chloride reference element and a platinum-wire indicating element in a single probe. This "dual element" configuration eliminates the need for two separate electrodes and is especially applicable to measurements in narrow-neck flasks and other restricted-entry receptacles.

The platinum combination electrode is recommended for use with automatic titrators and similar electroanalytical important. The close physical proximity of the porous-plug junction to the platinum-wire indicator section results it reduced resistance between these elements and

aduces a rapid dynamic response for both redox assurements and potentiometric titrations. Additionally, the electrode is ideally suited for any application that involves the measurement of oxidation-reduction potentials or requires the use of a "noble metal" sensor.

The electrode measures 5 inches with a 30-inch lead, and functions over a  $-5^{\circ}$  to 110°C temperature range. The filling solution is 4M KCI saturated with AgCI (Fisher No. So-P-135), and flow rate at the junction is less than 8  $\mu$ I per hour at an 8 cm head. Reference output is 44 $\pm$  ImV vs. S.C.E., while junction resistance is less than 10K ohms.

#### INSTALLATION

Place the platinum combination electrode into service as follows:

- 1. Remove cap from supplied filling-solution bottle, and screw on dispenser spout.
- 2 Lower rubber sleeve on electrode body until filling hole is xposed, and fill reference cavity with electrolyte until meniscus reaches a level approximately ¼-inch below filling hole.
- NOTE: Always use 4M KCl solution saturated with AgCl (Fisher No. So-P-135) as the electrolyte. NEVER USE SATURATED KCl FILLING SOLUTION.
- Place electrode upright in empty beaker to permit filling solution to wet and flow through porous plug, as evidenced by formation of KCI crystals on outer surface of plug.

NOTE: If no flow is observed within 30 minutes, or if response is unsatisfactory during an analysis, soak electrode in dilute KCI (0.1M) for several hours, and then perform the following procedure:

- a. Hold electrode (cap up) at a 45° angle between thumb and forefinger on left hand, so that filling hole faces out and is directly opposite base of thumb.
- b. Insert dispensing spout into filling hole.
- c. Make sure that electrode is supported by base of thumb, then firmly press spout into filling hole to make an airtight seal.

NOTE: Normally, spout tip will not touch internal element; while applying pressure, however, care should be exercised to prevent contact. If necessary, cut off a portion of the tip.

d. While maintaining seal, squeeze filling bottle firmly so

that electrode becomes pressurized.

NOTE: A bead of liquid should form at liquid junction in about 30 seconds; in some cases, however, it may be necessary to maintain pressure for several minutes. If flow cannot be established, refer to REJUVENATION section.

 Mount electrode on suitable holder and connect jacks to pH meter.

#### **OPERATION**

For optimumoperation with the platinum combination electrode, observe the following general procedures:

- Rubber sleeve should always be lowered on electrode body to expose filling hole and permit proper electrolyte leakage.
- Level of electrolyte must always be maintained above surface of sample solution to avoid backflow of sample into electrolyte. Refill reference cavity as required.
- After removing electrode from one solution and before immersing in another, the outer surface should be rinsed with distilled water.

#### STORAGE

When not in use, store the platinum combination electrode as follows:

- 1. Slide rubber sleeve into position over the filling hole.
- Place supplied cot over tip of electrode by threading platinum wire through opening and sliding cot onto glass body until porous plug is completely covered.

#### **REJUVENATION**

Rejuvenation of the platinum combination electrode may only require a simple cleaning. Occasionally, a more thorough cleaning is required, or the porous-plug junction may have to be unblocked. Each is covered separately below

#### Simple Cleaning

A simple cleaning of the electrode is done as follows:

- Wash electrode surface with a good detergent.
   NOTE: RBS-25 detergent (Fisher No. So-C-181) is recommended.
- 2. Polish platinum wire with scouring powder.
- 3. Rinse electrode thoroughly with distilled water.

#### Thorough Cleaning

For a more thorough cleaning, perform the following:

- Connect tip of large cable plug to negative terminal of a 22V dry cell, then immerse tip of electrode in a 1N solution of hydrochloric acid.
- Similarly connect a platinum or graphite electrode to positive terminal of dry cell and immerse tip of electrode in same solution.

NOTE: Hydrogen will evolve rapidly, and the metallic electrode will be cleaned by electrolysis in 5 to 10 seconds.

3. After cleaning, disconnect both electrodes and rinse



#### each with distilled water.

#### Unblocking the Junction

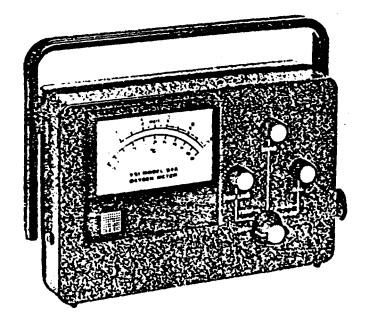
If the liquid junction should become partially blocked, perform the following:

- 1. Inspect reference cavity for crystallization.
- 2. If crystals are evident, proceed as follows:
  - Remove filling solution by shaking it out through filling hole.
  - b. Rinse cavity repeatedly with distilled water until all crystals are disolved.
  - Refill cavity with fresh 4M KCl solution saturated with AgCl (Fisher No. So-P-135).
    - CAUTION: Never use saturated KCI as the electrolyte.
  - d. Řepeat all of step 3 under INSTALLATION.
- 3. If difficulty persists, perform the following in sequence depending upon the severity of the blockage:
  - a. Soak electrode overnight in dilute KCl (0.1M).
  - b. Boil junction in dilute KCI for 5 to 10 minutes.
  - c. Carefully sand or file the porous plug junction.



APPENDIX B3
DISSOLVED OXYGEN FIELD SAMPLING SOP

# INSTRUCTION MANUAL YSI MODELS 54ARC AND 54ABP DISSOLVED OXYGEN METERS







Scientific Division
Yellow Springs Instrument Co., Inc.
Yellow Springs, Ohio 45387, U.S.A. • Phone 513-767-7241

PRICE INCLUDING HANDLING \$5.00

#### SUMMARY OF OPERATING INSTRUCTIONS

#### 1. CALIBRATION

- A. Switch instrument to OFF and adjust meter mechanical zero.
- B. Switch to RED LINE and adjust.
- C. Prepare probe for operation, connect to instrument, wait up to 15 minutes for probe to stabilize. Probe can be in calibration chamber or ambient air.
- D. Switch to ZERO and adjust to "0" on mg/l scale.
- E. Switch to TEMP and read on °C scale.
- F. Use probe temperature and true local atmospheric pressure (or feet above sea level) to determine calibration values from Tables I and II. (See pages 14 and 15).

EXAMPLE: Probe temperature = 21°C; Altitude = 1000 feet. From Table 1 the calibration value for 21°C is 8.9 mg/l. From Table II the altitude factor for 1000 feet is approximately .96. The correct calibration value, then, is:

 $8.9 \text{ mg/I} \times .96 \text{ factor} = 8.54 \text{ mg/I}$ 

G. Switch to 0-10 or 0-20 mg/l range and adjust meter with CAL control to calibration value determined in Step F.

NOTE: It is desirable to calibrate probe in a high humidity environment. (See calibration section for more detail).

#### 2. MEASUREMENT

- A. Place probe in sample and stir.
- Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.
- C. Read dissolved oxygen on appropriate range (1-10 or 0-20 mg/l)
- D. We recommend the instrument be left on between measurements to avoid the necessity to repolarize the probe.

#### 3. GENERAL CARE

- A. Recharge batteries in the YSI Model 54ARC when the instrument can no longer be red lined. Recharge 16-20 hours. Replace with Burgess CD-6 or equivalent. Replace batteries in the YSI Model 54ABP when red line cannot be set with Panasonic UM-2N or equivalent.
- B. Membranes will last indefinitely, depending on usage. Average replacement is 2-4 weeks. Probe should be stored in humid environment to prevent drying out.
- C. Calibrate daily.

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#### **GENERAL DESCRIPTION**

The YSI Models 54ARC and 54ABP Dissolved Oxygen Meters are interior dissolved oxygen and temperature measurement in water and wastewater pplications, but are also suitable for use in certain other liquids. Dissolved Oxygen is indicated in mg/l (milligrams per liter) on 0-10 and 0-20 mg/l scales, emperature is indicated in °C on a -5° to +45°C scale. Both dissolved oxygen anges are automatically temperature compensated for solubility of oxygen in vater and permeability of the probe membrane.

The probes use Clark-type membrane covered polarographic sensors with built-in thermistors for temperature measurement and compensation. A thin, permeable membrane stretched over the sensor isolates the sensor elements from the environment, but allows oxygen and certain other gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

The membrane passes oxygen at a rate proportional to the pressure difference across it. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure inside the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the absolute pressure of oxygen outside the membrane. If the oxygen pressure increases, more oxygen diffuses through the membrane and more current flows through the sensor. A lower pressure results in less current.

Power to operate the system is provided by internal batteries in the instruments, rechargeable batteries in the YSI Model 54ARC and disposable batteries in the YSI Model 54ABP.

#### **SPECIFICATIONS**

#### 1. Instrument

#### Oxygen Measurement

Ranges: 0-10 and 0-20 mg/l (0-5 and 0-10 mg/l with YSI 5776 High

Sensitivity Membrane)

Accuracy: ±1% of full scale at calibration temperature (±0.1 mg/l and

0-10 scale).

Readability: .05 mg/l on 0-10 scale; 0.1 mg/l on 0-20 scale.

#### Temperature Measurement

Ranges: -5° to +45°C

Accuracy: ± 0.7°C, including probe

Readability: 0.25°C

#### Temperature Compensation

 $\pm$  1% of D.O. reading for measurements made within  $\pm\,5^{\circ}\text{C}$  of calibration temperature.

±3% of D.O. reading over entire range of -5 to +45°C Probe temperature.

#### System Response Time

Typical response for temperature and D.O. readings is 90% in 10 seconds at constant temperature of 30°C with YSI 5775 Membranes. D.O. response at low temperature and low D.O. is typically 90% in 30 seconds. YSI 5776 High Sensitivity Membranes can be used to improve response at

low temperature and low D.O. concentrations. If response time under any operating conditions exceeds minutes, probe service is indicated.

#### **Operating Temperature Range**

Instrument and probe operating range is -2° to +45°C. Large ambient temperature changes will result in 2% loss of accuracy unless Red Line and Zero are reset.

#### Recorder Output

0 to 114-136 mV. Recorder should have 50,000 ohms minimum input impedance.

#### **Power Supply**

YSI Model 54ABP: (4) 1.5 volt carbon zinc batteries provide approximately 1000 hours operation. Replace with Panasonic UM-2N or equal.

YSI Model 54ARC: (4) 1.25 volt Ni-Cad rechargeable cells (Burgess CD-6 or equal) provide approximately 100 hours of operation between charges.

#### II. Probe

Cathode: Gold Anode: Silver

Membrane: .001" FEP Teflon (.0005" FEP Teflon available)

Electrolyte: Half Saturated KCI

Temperature Compensation: (See SPECIFICATIONS, I. Instrument)
Pressure Compensation: Effective 1/2% of reading to pressures of 100 psi

(230 ft. water)

Polarizing Voltage: 0.8 volts nominal

Probe Current: Air at 30°C = 19 microamps nominal
Nitrogen at 30°C = .15 microamps or less

#### III. Accessories and Replacement Parts

YSI 5720A — Self Stirring B.O.D. Bottle Probe

YSI 5750 - Non Stirring B.O.D. Bottle Probe

YSI 5739 — Oxygen Temperature Probe for field use. Combine with one of the following cables for desired lead length:

YSI 5401 — Battery Charger Eliminator 115V

YSI 5402 — Battery Charger Eliminator 230V

#### Detachable leads for use with YSI 5739:

10' cable
25' cable
50' cable
100' cable
150' cable
200' cable

YSI 5492A — Battery Pack Operates YSI 5791A and 5795A Submersible Stirrers

- YSI 5791A Submersible Stirrer for field use
- YSI 5795A Submersible Stirrer for field use
- YSI 5075A Calibration Chamber for use with field probe
- YSI 5890 Carrying Case
- YSI 5775 Membrane and KCI Kit, Standard includes 2 each 15membrane packets (.001" thick standard membranes) and a 30 ml bottle KCI with Kodak Photo Flo.
- YSI 5776 Membrane and KCI Kit, High Sensitivity includes 2 each 15-membrane packets (.0005" thick membranes) and a 30 ml bottle. KCI with Kodak Photo Flo.
- YSI 5945 "O" Ring Pack includes (6) "O" rings for each YSI D.O. Probe.
- YSI 5486 Beater Boot Kit includes (1) A-05486 Boot, (1) A-05484 Tip, (2) A-05485 Spring. Used only on 5720A and discontinued 5420A and 5720.
- YSI 5986 Diaphragm Kit for use only with YSI 5739 D.O. Probe.
- YSI 5734 Adaptor makes it possible to use discontinued YSI 5400 Series Probes with YSI Models 54ARC and 54ABP.
- YSI 5735 Adaptor makes it possible to use YSI 5739, 5720A and 5750 Probes with discontinued YSI Models 54RC and 54BP.

#### OXYGEN PROBES AND EQUIPMENT

There are three oxygen probes for use with the YSI Models 54ARC and 54ABP Dissolved Oxygen Meters. Descriptions of where they are used are contained in the following paragraphs.

#### 1 VSI 5739 D.O. Probe

The YSI 5739 probe, with built-in lead weight and pressure compensation, is an improved design that replaces the discontinued YSI 5418, 5419, 5718 and 5719 probes. (See Figure 1)

For user convenience the probe is equipped with a disconnecting cable to facilitate changing cable lengths and replacing damaged cables or probes. The probe and cable assembly is held together with a threaded retaining nut. The connection is *not* designed for casual disconnection and should only be disconnected when necessary.

To disconnect the cable unscrew the retaining nut and slide it down the cable to expose the connector. Pull gently on the cable and connector until the connector comes away from the probe body.

To reassemble, inspect the connector and "O" ring for cleanliness. If the "O" ring is frayed or damaged remove it by squeezing it in the groove causing it to bulge, then roll it out of the groove and off the connector. A replacement "O" ring is supplied with the cable.

Push the connector into the probe body, rotating it until the two halves mate. A light coating of vaseline or silicone grease on the "O" ring will make reassembly easier. Air trapped between the connector halves which may cause them to spring apart slightly, is normal. Screw on the retaining nut, hand tight only. NOTE: If erratic readings are experienced, disconnect the cable and inspect for water. If present, dry out and reconnect, replacing the "O" ring, if necessary.

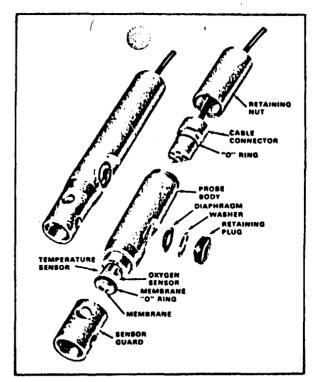


Figure 1

#### Pressure Compensation

The vent on the side of the probe is part of a unique pressure compensating system that helps assure accurate readings at great depths of water. Pressure compensation is effective to 1/2% of reading with pressures to 100 psi (230 ft. water). The quantity of air bubbles trapped under the membrane determines how serious the pressure error will be, which is why proper preparation of the probe is essential. (See OPERATING PROCEDURES.) The system is designed to accommodate a small amount of trapped air and still function properly, but the amount should be kept to a minimum

The compensating system normally does not require servicing and should not be taken apart. However, if electrolyte is leaking through the diaphragm or if there is an obvious puncture, the diaphragm must be replaced. A spare is supplied with the probe. Using a coin unscrew the retaining plug and remove the washer and the diaphragm, flush any salt crystals from the reservoir, install the new diaphragm (convolution side in), replace the washer, and screw in the retaining plug.

#### II. YSI 5720A B.O.D. Bottle Probe

The YSI 5720A B.O.D. Bottle Probe replaces the discontinued YSI 5420A B.O.D. Bottle Probe for measuring dissolved oxygen and temperature in standard B.O.D. bottles. It is provided with an agitator for stirring the sample solution, available in models for 117VAC (95-135VAC, 50-60 Hz) or 230VAC (190-250VAC, 50-60 Hz) operation. (See Figure 2)

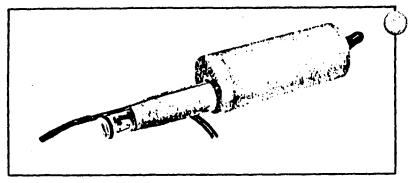


Figure 2

When using the probe, plug the agitator power supply into line power and the probe plug into the instrument. With the agitator turned off place the tapered probe end into the B.O.D. bottle and switch agitator "ON" with switch on top of probe. The probe should be operated with a minimum of trapped air in the B.O.D. bottle. A slight amount of air in the unstirred region at the top of the bottle may be neglected, but no bubbles should be around the thermistor or oxygen sensor.

#### Stirrer Boot

The probe uses a flexible stirring boot to transmit motion from the sealed motor housing to the sample. If the boot shows signs of cracking or other damage likely to allow leaking into the motor housing, the boot must be replaced.

In fresh water applications boot life is normally several years, but this may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life rinse the boot after use in contaminated samples. (See Figure 3)

Boot replacement is as follows:

- 1. Pull off old assembly and clean shaft.
- Slide on new assembly making sure the back spring is on the grooved area of the shaft. A small amount of rubber cement may be used.
- Check that there is sufficient clearance between the tip and the end of the shaft to permit turning without binding.

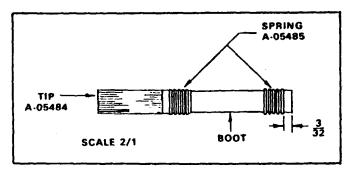


Figure 3

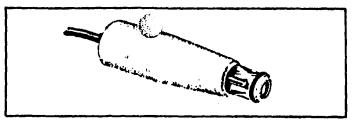


Figure 4

#### III. YSI 5750 B.O.D. Bottle Probe

The YSI 5750 B.O.D. Bottle Probe replaces the discontinued YSI 5450 B.O.D. Bottle Probe. It is similar to the YSI 5720A B.O.D. Bottle Probe, except that it does not have a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer. (See Figure 4)

#### IV. Cable Adaptors

All YSI 5700 Series Probes are designed for direct use with the YSI Models 54ARC and 54ABP Dissolved Oxygen Meters. However, to use YSI 5700 probes with the discontinued YSI Models 54RC and 54BP, cable adaptor YSI 5735 is required.

#### V. YSI 5791A and 5795A Submersible Stirrers

The YSI submersible stirrers are accessories that perform the function of stirring the sample being studied when making dissolved oxygen measurements in the field. The YSI 5791A stirrer can be used with the following dissolved oxygen probes: YSI 5418, 5419, 5718, 5719, and 5739. The YSI 5795A stirrer is only for use with the YSI 5739 Probe. (See Figure 6)

When a stirrer and probe are assembled, the stirrer agitates the sample directly in front of the sensor by means of a rotating eccentric weight which causes the spring-mounted hermetically sealed motor housing to vibrate. An impeller on the end of the motor housing flushes the media across the oxygen sensor. (See sales literature and instruction sheets for further information).

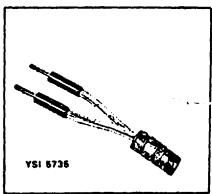


Figure 5

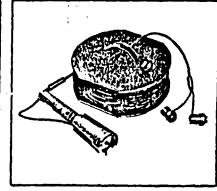


Figure 6

#### VI. YSI 5492A Battery Pack

The YSI 5492A Battery Pack is designed to attach to the case of YSI Model 54 Dissolved Oxygen Meters to provide power for operating the submersible stirrers. (See sales literature and instruction sheets for further information).

#### **OPERATING PROCEDURES**

#### 1. Preparing the Probe

All YSI 5700 Series Probes have similar sensors and should be cared for in the same manner. They are precision devices relying on good treatment if high accuracy measurements are to be made. Prepare the probes as follows. (See Figure 7)

ALL PROBES ARE SHIPPED DRY — YOU MUST FOLLOW THESE INSTRUCTIONS

- Prepare the electrolyte by dissolving the KCI crystals in the dropper bottle with distilled water. Fill the bottle to the top.
- 2. Unscrew the sensor guard from the probe (YSI 5739 only) and then remove the "O" ring and membrane. Thoroughly rinse the sensor with KCI solution.
- 3. Fill the probe with electrolyte as follows:
  - A. Grasp the probe in your left hand. When preparing the YSI 5739 probe the pressure compensating vent should be to the right. Successively fill the sensor body with electrolyte while pumping the diaphragm with the eraser end of a pencil or similar soft, blunt tool. Continue filling and pumping until no more air bubbles appear. (With practice you can hold the probe and pump with one hand while filling with the other.) When preparing the YSI 5720A and 5750 probes, simply fill the sensor body until no more air bubbles appear.
  - B. Secure a membrane under your left thumb. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, keeping it clean and dust free, touching it only at the ends.
  - C. With the thumb and forefinger of your other hand, grasp the free end of the membrane.
  - D. Using a continuous motion stretch the membrane UP, OVER, and DOWN the other side of the sensor. Stretching forms the membrane to the contour of the probe. The membrane can be stretched to approximately 1-1/2 times its normal length.
  - E. Secure the end of the membrane under the forefinger of the hand holding the probe.
  - F. Roll the "O" ring over the end of the probe. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the "O" ring.
  - G. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.
- 4. Shake off excess KCI and reinstall the sensor guard.
- 5. A bottomless plastic bottle is provided with the YSI 5739 probe for convenient storage. Place a small piece of moist towel or sponge in the bottle and insert the probe into the open end. This keeps the electrolyte from dry-

- ing out. The YSI 572^ and 5750 probes can be stored in a B.O.D. bottle containing about 1" was
- 6. Membranes will last indefinitely, depending on usage. Average replacement is 2-4 weeks. However, should the electrolyte be allowed to evaporate and an excessive amount of bubbles form under the membrane, or the membrane become damaged, thoroughly flush the reservoir with KCl and install a new membrane.
- Also replace the membrane if erratic readings are observed or calibration is not stable.
- "Home brew" electrolyte can be prepared by making a saturated solution of reagent grade KCI and distilled water, and then diluting the solution to half strength with distilled water. Adding two drops of Kodak Photo Flo per 100 ml of solution assures good wetting of the sensor, but is not absolutely essential.
- 9. The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), return it to the factory for service. Never use chemicals or any abrasive.
- 10. H2S, SO<sub>2</sub>, Halogens, Neon, Nitrous Oxide and CO are interfering gases. If you suspect erroneous readings, it may be necessary to determine if these are the cause. These gases have been tested for response.

100% Carbon Monoxide-Less than 1% 100% Helium-none

100% Carbon Dioxide-Around 1%

100% Chlorine-2/3 O2 response

100% Nitrous Oxide-1/3 Oz response

100% Hydrogen-Less than 1%

100% Nitric Oxide-1/3 Oz response

100% Ethylene-none

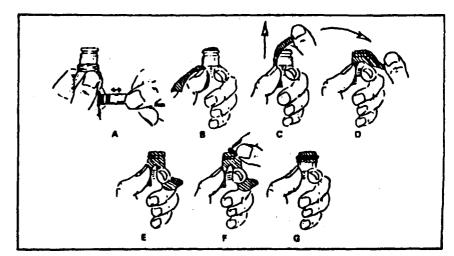


Figure 7

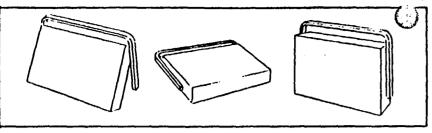


Figure 8

#### II. Preparing the Instrument

It is important that the instrument be placed in the intended operating position vertical, tilted, or on its back — before it is prepared for use and calibrated. (See Figure 8). Readjustment may be necessary when the instrument operating position is changed. After preparing the probe proceed as follows:

- With switch in the OFF position, adjust the meter pointer to Zero with the screw in the center of the meter panel. Readjustment may be necessary if the instrument position is changed.
- 2. Switch to RED LINE and adjust the RED LINE knob until the meter needle aligns with the red mark at the 31°C position.
- 3. Switch to ZERO and adjust to zero with zero control knob.
- 4. Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight.
- Before calibrating allow 15 minutes for optimum probe stabilization.
   Repolarize whenever the instrument has been OFF or the probe has been disconnected.

#### III. Calibration

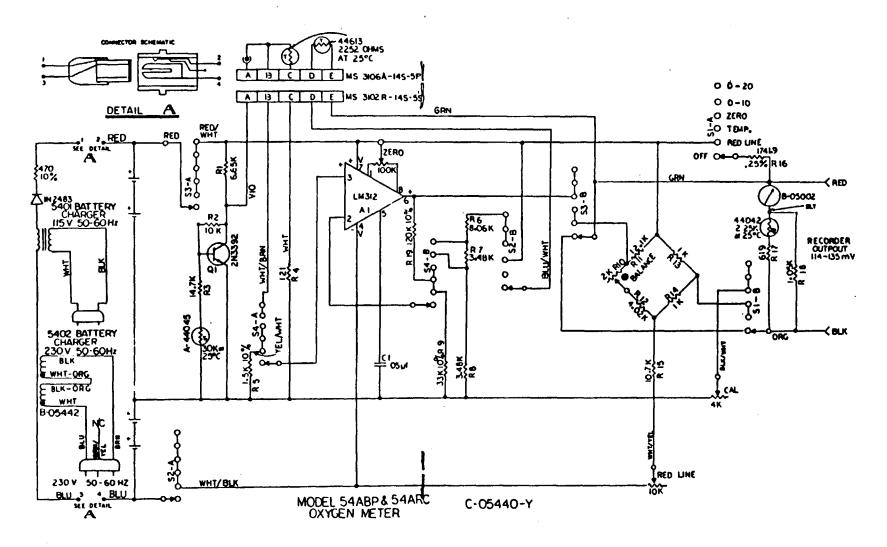
The operator has a choice of three calibration methods — Winkler Titration. Saturated Water, and Air. Experience has shown that air calibration is quite reliable, yet far simpler than the other two methods. The three methods are described in the following paragraphs.

#### Winkler Titration

- Draw a volume of water from a common source and carefully divide into four samples. Determine the oxygen in three samples using the Winkler Titration technique and average the three values. If one of the values differs from the other 2 by more than 0.5 mg/l, discard that value and average the remaining two.
- 2. Place the probe in the fourth sample and stir.
- 3. Switch to desired mg/I range and adjust the CALIBRATION control to the average value determined in Step 1. Allow the probe to remain in the sample for at least two minutes before setting the calibration value, and leave in the sample for an additional 2 minutes to verify stability. (Readjust if necessary).

#### Saturated Water

 Air saturate a volume of water (300-500 cc) by aerating or stirring for at least 15 minutes at a relatively constant temperature.



#### GENERAL HOTES:

- 1. ALL RESISTOR VALUES ARE IN GOOS; E-1,000, N-1,000,000. UNLESS OTHERMISE SPECIFIED, RESISTORS ARE 1/4W, 1% NETAL FILM.
- 2. FOR SAMP MATTERIES ARE 4 EA 1.5V PAMASONIC UN-2N ON EQUIV. FOR SAMPC MATTERIES ARE 4 EA 1.25V BURGESS COS OR (QUIV. NICADO.
- 3. BATTERY CHARGERS AND CONNECTOR APPLY TO SAARC YERSION ONLY.
- 4. THE VALUES SHOWN ON THE SCHEMATIC MAY DIFFER FROM THOSE IN THE INSTRUMENT; IF SO, ETTHER VALUE CAN BE USED FOR REPLACEMENT PURPOSES.

- 2. Place the probe in the sample and stir. Switch to TEMPERATURE. Refer to Calibration Table I for place of value corresponding to the temperature.
- 3. Determine Incal altitude or the "true" atmospheric pressure (note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level). Using Calibration Table II determine the correction factor for your pressure or altitude.
- 4. Multiply the mg/I value from Table I by the correction factor from Table II to determine the corrected calibration value for your conditions.
  - EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/l. From Table II the correction factor for 1000 feet is about 0.96. The corrected calibration value is 8.9 mg/l X 0.96 = 8.54 mg/l.
- Switch to an appropriate mg/l range and adjust the CALIBRATE knob while stirring until the meter reads the corrected calibration value from Step 4. Leave the probe in the sample for two minutes to verify calibration stability. Readjust if necessary.

#### Air Calibration - Fresh Water

- 1. Place the probe in moist air. B.O.D. probes can be placed in partially filled (50 ml) B.O.D. bottles. Other probes can be placed in the YSI 5075A Calibration Chamber (refer to the following section describing CALIBRATION CHAMBER) or the small calibration bottle (the one with the hole in the bottom) along with a few drops of water. The probe can also be wrapped loosely in a damp cloth taking care the cloth does not touch the membrane. Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is stabilizing.
- 2. Switch to TEMPERATURE and read. Refer to Table I Solubility of Oxygen in Fresh Water, and determine calibration value.
- 3. Determine altitude or atmospheric correction factor using Table II.
- Multiply the calibration value from Table I by the correction factor from Table II.

EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/l. From Table II the correction factor for 1000 feet is about 0.96. Therefore, the corrected calibration value is 8.9 mg/l X 0.96 = 8.54 mg/l.

5. Switch to the appropriate mg/l range and adjust the CALIBRATE knob until the meter reads the corrected calibration value from Step 4. Wait two minutes to verify calibration stability.

Readjust if necessary.

#### Air Calibration - Sea Water

1. Place the probe in moist air. B.O.D. probes can be placed in partially filled (50 ml) B.O.D. bottles. Other probes can be placed in the YSI 5075A Calibration Chamber (refer to the following section describing Calibration Chamber) or the small storage bottle (the one with the hole in the bottom) along with a few drops of water. The probe can also be wrapped loosely in a damp cloth taking care the cloth does not touch the membrane. Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is polarizing.

# YSI 5700 SERIES DISSOLVED OXYGEN PROBES INSTRUCTIONS

The probes described in these instructions are designed for direct use with YSI Models 50, 51B, 54ABP, 54ARC, 56, 57 and 58 Dissolved Oxygen Meters. The probes can also be used with discontinued YSI Models 51A, 54BP and 54RC Dissolved Oxygen Meters when the YSI 5735 Cable Adapter is employed.

#### PRINCIPLES OF OPERATION

YSI 5700 Series Probes are polarographic sensors. A thin permeable membrane stretched over the sensor isolates the electrodes from the environment, but allows gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

The membrane passes oxygen at a rate proportional to the difference across it in partial pressure of oxygen. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure under the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the partial pressure of oxygen outside the membrane. As the oxygen partial pressure varies, both the oxygen diffusion through the membrane and the probe current will change proportionally.

#### SPECIFICATIONS

Cathode: Gold Anode: Silver

Hembrane: .001" FEP Teflon, standard Electrolyte: Half saturated KCl Temperature Range: -5° to 45°C

15° to 35°C for the 5760 probe

Temperature Accuracy: ±0.2°C

Temperature Compensation: (see instrument specifications)

Polarizing Voltage: 0.8 Volts (nominal)

Probe Current in Air at 30°C: 19 microamps (nominal) in Nitrogen at 30°C: 0.15 microamps or less

Response Time: Typical response for dissolved oxygen, using standard membranes, is 90% in 10 seconds at a constant temperature of 30°C.

Response at low dissolved oxygen levels is typically 90% in 30 seconds.

#### ACCESSORIES AND REPLACEMENT PARTS

YSI 5492A Battery Pack for Hodels 51B and 54A (Powers the submersible stirrers.)

YSI 5735 Cable Adapter (Mates 5700 Series probes with discontinued YSI Models 51A, 54BP and 54RC Dissolved Oxygen Meters)

Accessories for the 5720A, 5739 and 5750

YSI 5680 Probe Reconditioning Kit. Includes a sanding tool and ten adhesive disks.

YSI 5775 Hembrane and KCl Kit, Standard. Includes two 15-membrane packets (.001" thick standard FEP Teflon membranes) and a 30 ml bottle of KCl with Kodak Photo Flo.

YSI 5776 Membrane and KCl Kit, High Sensitivity. Includes two 15-membrane packets (.0005" thick FEP Teflon membranes) and a 30 ml bottle of KCl with Kodak Photo Flo. Used for measurements below 15°C and/or for low oxygen levels

YSI 5793 .001" membranes, 10-membrane packet

YSI 5794 .0005" .embranes, 10-membrane packet

YSI 5945 O-ring pack (Contains replacement sensor O-rings)

Accessories for the 5720A Only

YSI 5486 Stirrer Boot Assembly

Accessories for the 5739 Only

YSI 5075A Calibration Chamber YSI 5986 Diaphragm Kit

YSI 5740-10 detachable 10' cable

YSI 5740-25 detachable 25' cable YSI 5740-50 detachable 50' cable

YSI 5740-100 detachable 100' cable

YSI 5740-150 detachable 150' cable

YSI 5740-200 detachable 200' cable

YSI 5791A Submersible Stirrer with 50' cable for stirrer only

YSI 5795A Submersible Stirrer with 50' combined probe and stirrer cable

#### YSI 5720A BOD BUTTLE PROBE

The 5720A bottle probe (Figure 1) is used for measuring dissolved oxygen in standard BOD bottles. It is provided with a stirrer powered by a DC supply available for 115 or 230 VAC input.

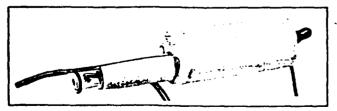


Figure 1. The YSI 5720A Probe

Yellow Springs Institument Co., Inc., Yellow Springs, Ohio 45387 USA • Phone 513 767-7241 • 800 343-HELP • Fax 513 767-9353 • Telex 205437 •

- 2. Switch to TEMPERATURE and read. Refer to Table III Solubility of Oxygen in Sea Water, and determine calibration value.
- 3. Switch to the appropriate mg/l range, and adjust the CALIBRATE knob until the meter reads the calibration value determined in Step 2. Wait 2 minutes to verify calibration stability. Readjust if necessary.

The probe is now calibrated and should hold this calibration value for many measurements. Calibration can be disturbed by physical shock, touching the membrane, or drying out of the electrolyte. Check calibration after each series of measurements and in time you will develop a realistic schedule for recalibration. For best results when not in use, follow the storage procedures recommended for the various probes described under OXYGEN PROBES AND EQUIPMENT. This will reduce drying out and the need to change membranes.

#### Calibration Chamber

The YSI 5075A Calibration Chamber is an accessory that helps obtain optimum calibration in the field and is also a useful tool for measuring at shallow depths (less than 4').

As shown in Figure (A), it consists of a 4-1/2 foot stainless steel tube (1) attached to the calibration chamber (2), the measuring ring (3), ...d two stoppers (4) and (5).

For calibration, insert the solid stopper (4) in the bottom of the calibration chamber (2). Push the oxygen probe (6) through the hollow stopper (5) as shown in Figure (B). Place the probe in the measuring ring, Figure (C), and immerse the probe in the sample to be measured for five minutes to thermally equilibrate the probe. Quickly transfer the probe to the calibration chamber (5) draining excess water from the chamber and shaking any excess droplets from

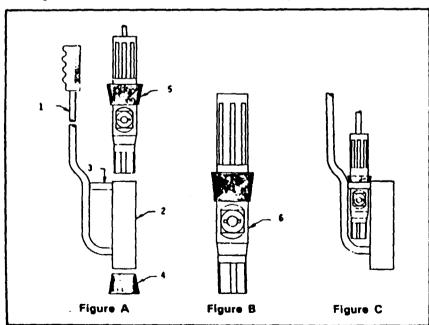


Figure 9

the probe membrane. For maximum accuracy, wet the inside of the calibration chamber with fresh water. This es a 100% relative humidity environment for calibration. Place the chamber in the sample for an additional five minutes for final thermal equilibrium. Calibrate the probe as described in the aircalibration procedure. Keep the handle above water at all times.

After calibration, return the probe to the measurement ring for shallow measurements. Move the probe up and down, or horizontally, approximately one foot a second while measuring. In rapidly flowing streams (greater than 5'/second) install the probe in the measuring ring with the pressure compensating diaphragm towards the chamber.

#### IV. Dissolved Oxygen Measurement

With the instrument prepared for use and the probe calibrated, place the probe in the sample to be measured and provide stirring.

- Stirring for the YSI 5739 Probe can best be accomplished with a YSI submersible stirrer. If the submersible stirrer is not used, provide manual stirring by raising and lowering the probe about 1 ft. per second. If the 5075 Calibration Chamber is used, the entire chamber may be moved up and down in the water at about 1 ft. per second.
- 2. The YSI 5720A has a built-in power driven stirrer.
- 3. With the YSI 5750 sample stirring must be accomplished by other means such as with the use of a magnetic stirring bar.
- Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.
- 5. Read dissolved oxygen.

#### V. High Sensitivity Membrane

Use of high sensitivity .0005" membranes (YSI 5776) in place of standard .001" membrane (YSI 5775) is recommended when measurements are to be made consistently at low temperatures (less than 15°C). Calibration and readings will be made just as if the standard YSI 5775 Membrane was being used.

The YSI 5776 High Sensitivity Membranes can also be used in certain situations to increase sensitivity at temperatures about 15°C. The ranges thus become 0-5 and 0-10 mg/l. When calibration with high sensitivity membranes is attempted at temperatures greater than 15°C the selector switch must be set to 0-20 mg/l. Multiply the calculated calibration value by 2. For example: at 21°C and 1000 ft. altitude the calibration value would be 8.6 x 2 or 17.2. Remember the 0-10 and 0-20 mg/l ranges are now 0-5 and 0-10 mg/l, and all mg/l readings must be divided by 2 for a final reading. When operating in this manner accuracy will be degraded slightly.

#### VI. Recorder Output

Red and black recorder jacks are provided on the YSI Models 54ARC and 54ABP, if you wish to record data while measuring. The high terminal of the recorder is connected to the red tip jack and the low terminal to the black. Output of the YSI 54A at full scale is between 114 to 136 mV.

Use a 50K or higher input impedance recorder and operate it with the terminals ungrounded. The recorder should be operated with its terminals ungrounded. Calibration of the instrument should be checked after connection of the recorder.

Many recorders have an adjustable full scale sunsitivity for those recorders are used with the Model 54A, use the 100 millivolt range adjust the full scale chart deflection when there is full scale meter deflection. Refer to the instruction book for the recorder. For recorders without this feature, a simple divider network as shown below can be constructed. This is adequate to adjust the signal for full scale chart and meter deflection on the 100 mV fixed range recorders.

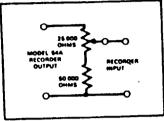


Figure 10

#### VIII. Calibration Tables

Table I shows the amount of oxygen in mg/I that is dissolved in air saturated fresh water at sea level (760 mmHg atmospheric pressure) as temperature varies from 0° to 45°C.

Table 1 — Solubility of Oxygen in Fresh Water

Temperature °C	mg/l Dissolved Oxygen	Temperature °C	mg/l Dissolved Oxygen
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
	11.27	33	7.16
10 11	11.01	34	7.05
	10.76	35	6.93
12 13	10.52	36	6.82
	10.29	37	6.71
14	10.07	38	6.61
15	9,85	39	6.51
16	9.65	40	· 6.41
17	9.45	41	6.31
18	9.26	42	6.22
19 20	9.07	43	6.13
20	8.90	44	6.04
21 22	8.72	45	<b>5.95</b>

Source: Derived from 15th Edition "Standard Methods for the Examination of Water and Wastewater."

#### Table II -- Correction ( Atgrapheric Pressure

Table II shows the correction to that should be used to correct the calibration value for the effects of atmospheric pressure or altitude. Find true atmospheric pressure in the left hand column and read across to the right hand column to determine the correction factor. (Note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level.) If atmospheric pressure is unknown, the local altitude may be substituted. Select the altitude in the center column and read across to the right hand column for the correction factor.

Table II

Atmospheric Pressure mmHg	equivalent Altitude or Ft.	= Correction Factor	
775	540	1.02	
760	0	1.00	
745	542	.98	
730	1094	.96	
714	1688	.94	
699	2274	.92	
684	2864	.90	
669	3466	.88	
654	4082	.86	
638	4756	.84	
623	5403	.82	
608	6065	.80	
593	6744	.78	
578	7440	.76	
562	8204	.74	
547	8939	.72	
532	9694	.70	
517	10472	.68	
502	11273	.66	

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

The temperature-solubility relationship of oxygen in sea water is not the same as that in fresh water. For this reason the compensation error where  $\frac{1}{2}$  and with sea water is greater than when used with fresh water. For a  $\pm 5^{\circ}$ C span the error could be  $\pm 2.2\%$  of reading and over the temperature range of  $\pm 2^{\circ}$  to  $\pm 30^{\circ}$ C the error could be 6.3% of reading.

Table III - Solubility of Oxygen in Sea Water

# SOLUBILITY OF OXYGEN IN SEA WATER (Chloride concentration 20,000 mg/1)

Temp. °C	Solubility mg/l	Temp. °C	Solubility mg/l
0	11.41	16	7.91
1	11.11	17	7.78
2	10.83	18	7.61
3	10.56	19	7.47
4	10.30	20	7.33
5	10.05	21	7.20
6	9.82	22	7.07
7	9.59	23	6.95
8	9.37	24	6.83
9	9.16	25	6.71
10	8.96	26	6.60
11	8.77	27	6.49
12	8.58	28	6.38
13	8.41	29	6.28
14	8.24	30	6.18
15	8.07		

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

#### Correcting for Salinity

When measuring dissolver oxygen in water samples with a salinity or chlorinity between sea water and fresh water, calibrate the instrument for fresh water and make your measurements. Then correct the data according to the following formula:

#### FORMULA:

$$A = M[1.0 - \frac{(Cs/Co[Si-So])}{Si}]$$

Where: A = Actual DO of sample. (mg/l dissolved O<sub>2</sub>)

M = Measured DO with instrument

\*Co = Chlorinity of ocean water (20 o/oo Cl ion)

'Cs = Chlorinity of sample (o/oo Cl ion)

Sf = DO of saturated fresh water at 760 mm pressure and at same temperature as sample (mg/l DO, obtain data from charts in instruction manual)

So = DO of saturated ocean water (20,000 mg/l Chloride ion) at 760 mm pressure and at same temperature as sample (mg/l DO, obtain data from instruction manual)

\*NOTE: If salinity is used instead of chlorinity the ratio Cs/Co is computed using 36.11 o/oo for Co (salinity of ocean water), and the salinity of your sample of Cs.

**EXAMPLE:** Measured Data

$$DO = 4.1$$

$$A = 4.1 [1.0 - (\frac{[31.0/36.11]}{8.8} [8.8 - 7.1])$$

$$= 4.1 [1.0 - (\frac{8.6}{8.8})]$$

$$= 4.1 [1.0 - (\frac{1.46}{8.8})]$$

$$= 4.1 [1.0 - 0.166]$$

$$= 4.1 [0.834]$$

$$= 3.41 \text{ mg/}$$

#### ISCUSSION OF MEASUREMENT ERRORS

There are three basic types of errors which can occur. Type I errors are related imitations of the instrument design and tolerances of the instrument compnents. These are chiefly the meter linearity and resistor tolerances. Type II rors are due to basic probe accuracy tolerances, chiefly background signal, robe linearity, and variations in membrane temperature coefficient. Type III rors are related to the operator's ability to determine the conditions at the time f calibration. If calibration is performed against more accurately known contions, Type III errors are appropriately reduced.

#### idividual Sources of Error

This description of sources of error can be used to attach a confidence to any articular reading of dissolved oxygen. The particular example given is for a near xtreme set of conditions. As a generality, overall error is diminished when the robe and instrument are calibrated under conditions of temperature and disolved oxygen which closely match the sample temperature and dissolved oxygen.

#### Type I

- is the error due to meter linearity
  Error = +1% full scale of the measurement range.
- 3 is the error due to tolerances in the instrument when transferring a reading from one range to another. Error = ±1% of the reading.

#### Type II

A - errors due to probe background current

Error = 1.0% (1 - Meter Reading mg/l Calibration Value mg/l

- B errors due to probe non-linearity. Error = ± 0.3% of reading.
- C error caused by variability in the probe membrane temperature coefficient.

Error = zero if readings are taken at the calibration temperature  $\pm$  1% of meter reading if readings are taken within 5°C of the

calibration temperature. Error  $= \pm 3\%$  of meter reading for all other conditions.

#### Type III

- A errors due to the accuracy of the instrument thermometer when used to measure the exact probe temperature during calibration.

  Error = ± 1.5% of reading.
- B errors due to the assumption of mean barometric pressure.
   Daily variation is usually less than 1.7%.
   Error = ±1.7% of reading.
- errors assume an ability to estimate altitude to within +500 ft. when computing the altitude correction factor.
   Error = ± 1.8% of reading.

D = errors consider the p = bility of only 50% relative humidity when calibrating the probe. It in a signal relative humidity is 50% instead of 100% the errors will be as follows:

Calibration Temperature ± C	Error in percent of reading
0 .	(-) 0.3
10	(·) 0.6
20	(-) 1.15
30	(-) 2.11
40	(•) 3.60

#### **Example of a Typical Error Calculation**

The example given presumes the air calibration technique. If calibration is done with air saturated water, the relative humidity consideration (III-D) is eliminated. If the Winkler calibration method is used, Type III errors are deleted and replaced by the uncertainty attributable to the overall Winkler determination.

Data: Instrument calibrated at 25°C, elevation estimated at 2000' ± 500', normal barometric pressure assumed, calibrated on 0-10 mg/l range at 7.8 mg/l, readings taken on 0-20 mg/l range at 10.5 mg/l at 8°C.

Туре	Description		Calculations		Error mg/l
IA	Linearity	-	.01 X 10.5 mg/l	=	.10
1B	Range Change	=	.01 X 10.5 mg/l	=	.10
	•		/ 10.5		
IIA	Probe Background	=	01 X \1 - 7.8 / 7.8 mg/l	=	.03
IIB	Probe Linearity	=	003 X 10.5 mg/l	=	.03
1IC	Temp. Compensation	=	03 X 10.5 mg/l	=	.31
IIIA	Temp. Measurement	=	.015 X 10.5 mg/l	=	.16
IIIB	Pressure	=	.017 X 10.5 mg/l	<b>=</b>	.18
IIIC	Altitude	=	.18 X 10.5 mg/l	=	.19
IIID	R.H.	=	.016 X 10.5 mg/l	=	.17
			Maximum Possible Error Probable Error	=	1.27 mg/l ± .63 mg/l

Considering a statistical treatment of the probable error at any time for any instrument, it is likely that the actual error in any measurement will be about 1/2 of the possible error. In this case the probable error is about  $\pm .5$  mg/l out of a reading of 10.5 mg/l, or 4.8% of the reading.

#### **INSTRUMENT BATTERIES**

Battery replacement or recharging on the YSI Model 54A is indicated if the "red line" adjustment cannot be made or O<sub>2</sub> calibration cannot be achieved. (Warning: a faulty probe will also not permit O<sub>2</sub> calibration.)

To replace batteries remove the four screws holding the rear cover of the instrument. The four batteries will be found on the battery terminal board inside. CAUTION: disconnect battery charger on YSI Model 54ARC before removing

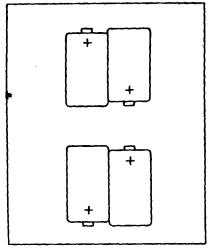


Figure 11

The YSI Model 54ARC contains four 1.25V Ni-Cd batteries (Burgess CD6 or equivalent). (See Figure 11). These batteries should be recharged when the instrument can no longer be red lined. Battery life should be three years or longer. Deeper discharge because of longer intervals between recharge will result in shorter battery life. The batteries should be recharged overnight, about 16 hours with the instrument off or 20 hours with the YSI Model 54ARC turned on.

The YSI Model 54ABP contains four 1.5V carbon-zinc (Panasonic UM-2N or equivalent). The life of these batteries is 1000 hours. Replace batteries every six months to minimize danger of corrosion due to dead or leaky batteries.

Battery holders are color coded. Positive (+ button) end of battery must go to red. (See Figure 11).

#### **WARRANTY AND REPAIR**

All YSI products carry a one-year warranty on workmanship and parts exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI dealer.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt servicing for all YSI products.

YELLOW SPRINGS INSTRUMENT CO., INC. SERVICE DEPARTMENT P.O. BOX 279
YELLOW SPRINGS, OHIO 45387, U.S.A.

PHONE: 513-767-7241

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To use the 5720A, plug the stirrer power supply into line power and the probe plug in the instrument. With the stirrer off, place the tapered probe end into a filled the BOD bottle and turn on the stirrer. The probe should be operated with a minimum of trapped air in the bottle. A slight amount of air in the unstirred region at the top may be neglected, but no bubble should be permitted around the sensor. CAUTION: The motor housing is not waterproof; do not submerge this probe beyond the part that is inserted into a BOD bottle.

#### Stirrer Boot (YSI 5486)

The 5720A uses a flexible stirring boot to transmit motion from the motor housing to the sample. If the boot shows signs of cracking or other damage liable to allow leakage into the motor housing, it must be replaced. Running the 5720A with a damaged stirring boot could cause permanent motor damage. Boot life may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life, rinse the boot after each use. Boots are replaced as follows:

- 1. Pull off the old assembly and clean the stir rod housing.
- 2. Slide on the new assembly, making sure the back spring is over the grooved area of the stir rod housing. A drop of alcohol will aid installation by providing lubrication.
- 3. Do not permit the stir rod to press against the end of the stirrer boot tip or it will bind.

#### YSI 5739 DISSOLVED OXYGEN PROBE

The 5739 probe system consists of the probe body plus a detachable cable (see Figure 2). The detachable cable is a convenience feature that facilitates changing cable lengths and replacing damaged cables or probes. The probe and cable assembly is held together with a threaded retainer. The assembly is not intended for casual disconnection; cable and probe should be separated only when necessary.

To detach the cable, unscrew the retainer and slide it down the cable to expose the connector. Pull gently on the connector until it comes away from the probe body. If the O-ring is frayed or damaged, replace it: a replacement O-ring is supplied with each 5740 cable. Reassemble by pushing the connector into the probe body, rotating it until the two halves mate. A light coating of silicone grease on the O-ring will make reassembly eatier. Be sure the connector is dry; otherwise, erratic readings may result. Screw on the retainer finger-tight only.

#### Pressure Compensation

The 5739 probe has a unique pressure compensating system that helps assure accurate readings at great depths. Pressure compensation is effective to 1/2% of reading with pressures up to 100 psi (230 feet of water). The compensating system does not normally require service and should not be taken apart. However, if electrolyte is leaking through the diaphragm, or if there is an obvious puncture, the diaphragm must be replaced. A spare is supplied with the probe. Use a coin to unscrew the retaining plug and remove the washer and diaphragm. With distilled water, flush any salt crystals from the reservoir, install a new diaphragm (flat side out), replace the washer and securely screw in the retaining plug.

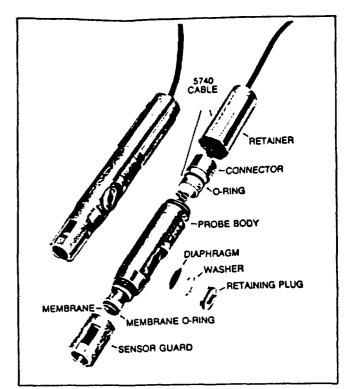


Figure 2. The YSI 5739 Probe

#### YSI 5750 BOD BOTTLE PROBE

The 5750 (Figure 3) is similar to the 5720A except that it does not have a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer.

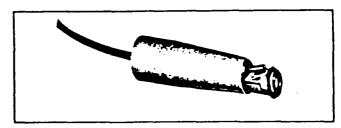


Figure 3. The YSI 5750 Probe

#### PRORE PREPARATION

All probes are shipped dry. You must follow these instructions when preparing a new probe or when changing membranes. Prepare the electrolyte by dissolving the KCl crystals which are supplied in a dropper bottle that should be filled to the neck with distilled water and shaken until the crystals are dissolved.

- 1. Unscrew the sensor guard (5739 only). Remove the O-ring and membrane, then thoroughly rinse the sensor with distilled water.
- 2. To fill the probe with electrolyte and install a new membrane, follow these steps:
- a. Grasp the probe in your left hand. (See the sketches in Figure 4.) When preparing the 5739 probe, the pressure compensating port should be to the right. Successively fill the sensor body with electrolyte while pumping the diaphragm with the eraser end of a pencil or a similar soft, blunt tool. Continue filling and pumping until no more

air bubbles appear. For ease in preparing the 5720A, the stirring rod should be to the left. When preparing the 5720A or 5750 probes, simply fill the sensor body until no more air bubbles appear.

- b. Secure a membrane between your left thumb and the probe body. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, touching it at the ends only.
- e. With the thumb and forefinger of your other hand, grasp the free end of the membrane.
- d. With a continuous motion, stretch it up, over and down the other side of the sensor. Stretching forms the membrane to the contour of the probe.
- Secure the end of the membrane under the forefinger of your left hand while holding the probe.
- f. Roll the O-ring over the end of the probe, being careful not to touch the membrane surface. For the 5720A, start at the right side of the sensor and roll the O-ring toward the stirring rod. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the O-ring.
- g. Trim off excess membrane with scissors or sharp' knife. Check that the stainless steel temperature sensor is not covered by excess membrane.
- 3. Shake off excess KCl. On the 5739, reinstall the sensor guard.

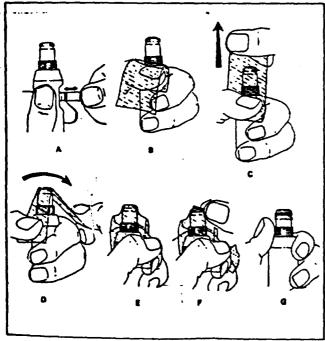


Figure 4. Membrane Application

#### Probe Storage

A bottomless plastic bottle is provided with the YSI 5739 probe for convenient storage. Place a small piece of moist towel or sponge in the bottle and insert the probe into the open end. This keeps the electrolyte from drying out. The 5720A and 5750 probes can be stored in a BOD bottle containing at least 1" of water.

#### OPERATING PRECAUTIONS. ALL PROBES

1. Membrane life depends on use. Membranes will last a long time if installed properly and treated with care during use. Erratic readings result from loose, wrinkled or fouled membranes, or from large bubbles in the electrolyte reservoir. If erratic readings, or evidence of membrane damage occur, you should replace the membrane and KCL. The average replacement interval is two to four weeks; electrolyte in constant or heavy use will be exhausted in about two weeks.

If the sensor O-ring on any probe is worn or loose, replace it with the O-ring provided in the YSI 5945 O-ring Pack.

- 2. The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), it needs to have its surface restored. Probes may either be returned to the factory, or cleaned with the YSI 5680 Probe Reconditioning Kit; never use chemicals or any abrasive not supplied with this kit.
- 3. It is also possible that the silver anode may become contaminated, which will prevent successful calibration. Try soaking the probe overnight in a 3% ammonia solution; rinse with deionized water, recharge with electrolyte, and install a new membrane. If still unable to calibrate after several hours, return the probe for service.
- 4. Hydrogen sulfide, sulfur dioxide, halogens, and meon are interfering gases. If you suspect erroneous readings, it may be necessary to determine if these are the cause.

These	gases	s have been teste	d for response:			
	100%	Carbon Monoxide		less	then	11
	100%	Carbon Dioxide		a	round	11
	100%	Hydrogen		less	than	11
	100%	Chlorine	2/3	0,	respon	154
	100%	Helium		4		วกจ
	100%	Nitrous Oxide	1/3	0-	respon	154
	100%	Ethylene		4		)De
	100%	Nitric Oxide	1/3	0-	788DO	

- 5. The correct liquid level in BOD bottles is achieved by overfilling, then inserting a stopper and pouring off the excess. When using a YSI 5760 or a 5720A probe in a filled BOD bottle, be careful to insert it slowly to avoid sample overflow.
- 6. When using the 5720A in samples containing heavy particulate solids, additional stirring may be needed. Inverting the stoppered bottle immediately before use will usually provide adequate mixing.

#### CALIBRATION

\_\_\_\_\_

Daily calibration is generally appropriate. Calibration can be disturbed by physical shock, touching the membrane, fouling of the membrane or drying out of the electrolyte. Check calibration after each series of measurements, and in time you will develop a realistic schedule for recalibration. When probes are not in use, store them as recommended in Probe Preparation.

Probes may be calibrated by Winkler Titration or by the Water Saturated Air method. Experience has shown that air calibration is quite reliable, yet far simpler than titration. Both methods are described here. Consult the manual for your particular instrument for more complete instructions.

#### Winkler Titration

- 1. Draw a volume of water from a single source andcarefully divide it into four samples. Determine the oxygen in three of the samples using the Winkler Titration technique and average the three values. If one of the values differs from the other two by more than 0.5 mq/L, discard it and average the two values remaining.
- 2. Using the probe-meter system you are calibrating, place the probe into the fourth sample and stir.
- 3. Switch to the desired mg/L range and adjust the CALIBRATION control to the average value determined in step 1. Allow the probe to remain in the sample for at least 5 minutes before setting the calibration value, then leave it in the sample for an additional two minutes to verify stability. Readjust if necessary.

#### Air Calibration

1. Place the probe in a BOD bottle containing about 1 Wait approximately ten minutes for inch of water. temperature stabilization.

The 5739 probe can be placed in the YSI 5075A Calibration Chamber or in the small calibration bottle supplied with the probe (the one with the hole in the bottom) along with a few drops of water, or a moistened towel or cloth.

2. Read the temperature and refer to the instrument Calibration Table to determine the calibration value. NOTE: To achieve the stated accuracy of measurement, the probe must be stabilized before calibrating. The calibration temperature should be within 5 degrees of the sample temperature.

- 3. Determine the atmospheric correction factor (see Instrument instructions).
- 4. Multiply the calibration value by the correction factor.
- 5. Switch your instrument to an appropriate mg/L range and adjust the CALIBRATE control until the meter reads the corrected calibration value from step 4. Without changing the calibration setup, monitor the readings for an additional 3 minutes to verify calibration stability. Readjust if necessary.

#### WARRANTY AND REPAIR

All YSI products carry a one-year warranty on workmanship and parts, exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI dealer. Electrode cleaning is not covered by warranty.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt servicing on all its products. This warranty is limited to repair or replacement (YSI's option) at no charge.

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APPENDIX B4

TOTAL ORGANIC CARBON LABORATORY ANALYSIS SOP

#### APPENDIX B4

#### QAPP ADDENDUM ACS PHASE II RI/FS

# TOTAL ORGANIC CARBON (TOC) ANALYSIS METHOD STANDARD OPERATING PROCEDURE

Determination of organic carbon (%) in soil, using sub-aliquots of air-dried soil, passed through a 100 mesh to 140 mesh screen. All of the sub-aliquot must pass the screen. Applicable organic carbon concentration range of interest is 0.1% to 2% (or larger) in soil, (dry weight basis). Laboratory may report lower concentration values.

Test procedures used for determining soil TOC concentrations shall be the 1) dry combustion (resistance furnace);2) dry combustion (induction furnace);3) dry combustion (automated methods), or 4) wet combustion (combustion train) methods of analysis specified by Table 29-1 of "Methods of Soil Analyses," Part 2 - Chemical and Microbiological Properties, 2nd ed., 1982, American Society of Agronomy, and Soil Science Society of America, Madison, Wisconsin. Copies of this copyrighted material are not being provided, because it is standard documentation at the laboratory to be used.

Any automated dry combustion test procedure used must provide results consistent with the other 3 methodologies and must be consistent with the requirements of Chapter 29, Sections 29-1, 29-2, and 29-3, "Methods of Soil Analysis" (MSA) Part II, 2nd ed., as appropriate. Soils can be calcareous or noncalcareous soils, with varying amounts of organic carbon. Soils determined may be subsurface as well as surface soils. If peat or muck soils are ever encountered, the laboratory will provide, with the case narrative, limitations of any sample results and any solutions to problems encountered. This is also true for any other problem sample types encountered.

The laboratory providing organic carbon analysis data, will provide a narrative discussion concerning methodology, instrumentation, and specific QA practices used for the set of soils tested. Requested information is detailed in items 2 and 3 of this SAS.

# ATTACHMENT 1 Analytical Methods - Organic Carbon in Soil

- 7a. Sample Preparation: Representative sub-aliquot of air-dried soil (see % solids SAS) screened through 100 or 140 mesh as appropriate. All of the sub-aliquot must pass this screen.
- b. Test for Presence of Inorganic Carbon, MSA, Part II, Section 29-3.3.1. Place finely ground soil on a spot plate, and moisten with a few drops of water. Add 4 N HCl dropwise to the wetted sample and observe any effervescence. Allow sufficient time for dolomite to react (-5 min). If inorganic carbon is absent proceed with Total Carbon in items #7c, or 7d below. If inorganic carbon is present, or the test is not definitive, proceed with items #7e, or #7f prior to Total Carbon measurements of Item #7c or #7d.
- c. Total Organic Carbon (Dry Combustion), MSA, Part II, Section 29-2.2.2. Use this as a guide for instrumental specifications. Instrument must test solid sample directly. Illustrative examples of this methodology are:
  - 1) Total Organic Carbon (Dry Combustion Medium Temperature Resistance Furnace), MSA, Part II, Section 29-2.2.3.
  - 2) Total Organic Carbon (Dry Combustion High Temperature Induction Furnace), MSA, Part II, Section 29-2.2.4.
  - 3) Total Organic Carbon (Dry Combustion Other Instrumental Methods), MSA, Part II, Section 29-2.2.5. Any other instrumentation such as this must be justified and provide results as precise and accurate as the results from Sections 29-2.2.3, and 29-2.2.4.
- d. Total Carbon (Wet Digestion), MSA, Part II, Section 29-2.3.2 Soil digested in 60:40 mixture of sulfuric acid and phosphoric acid (containing  $K_2CrO_7$ ).  $CO_2$  evolved is absorbed and weighed, or absorbed in standard base and titrated.
  - 1) Specific examples are found in MSA, Part II, Figure 29-2, Figure 29-3, and Section 29-2.3.3.
- e. Pretreatment prior to Dry Combustion, MSA, Part II, Section 29-3.3.3. Inorganic carbon is removed by treating sample in a combustion boat, with 5% sulfurous acid (H<sub>2</sub>SO<sub>3</sub>). After several hours, remove the excess H<sub>2</sub>SO<sub>3</sub> by leaving overnight in an evacuated dessicator. Read citation for further details.
- f. Pretreatment prior to Wet Digestion, MSA, Part II, Section 29-3.3.2. Inorganic carbon is removed by sulfuric acid ferrous sulfate reagent in apparatus used for total carbon (Wet Digestion) prior to Total Carbon measurement. See citation for further details.

- g. Use only the methods specified above or obtain approval of CPMS, CRL prior to use of other method. Test procedure description, and description of specific measurement principles, including equivalency to each of the 10 items of Figure 29-1 of MSA, Part II and sample pretreatment of Section 29-3, MSA, Part II, must be included with each set of soils tested.
- h. Laboratory performing Total Organic Carbon determinations must use and have a recognized procedure for removal of any inorganic carbon in sample.

#### ATTACHMENT 2

A variety of apparatus, instrumentation, sample preparation systems and read-outs can be used. It is the responsibility of the laboratory to provide appropriate QC audits and QC data with each set of samples tested.

If instrumentation requires calibration, provide calibration curve, including zero concentration standard and preparation blanks. Provide positive control (a test sample prepared independently from calibration standards) that provides a measure of accuracy of system. This should be done for all systems including grametric readouts.

# ATTACHMENT 3 Analytical Results Required

As part of Case Narrative, attach description of test procedure and instrumentation used for measurement of Total Organic Carbon and removal of any Inorganic Carbon. Test procedure description must include sufficient information that the nature of specific analytical result deliverables can be determined including QC audits. In Case Narrative, discuss any problem type samples (including peat or muck soils), limitations on any sample results, and solution taken to resolve any problems. A sample preparation log will be provided, as appropriate.

Bench record tabulating any order of any sample weights and tare weights of absorbed  $\rm CO_2$ , instrument calibrations, blanks, QA audits, etc., must be provided along with copies of any worksheets used to calculate results. Include copies of any instrument readouts. All must be legible. Report results as % of organic carbon on a dry weight basis (103-105°).

VACS ATTACHMENT/dms/GEP